Michigan Department of Environmental Quality

API/PC/KR Superfund Site
Time Critical Removal Action Monitoring
Quality Assurance Project Plan

January 2008

Final Report

US EPA RECORDS CENTER REGION 5
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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

January 23, 2008 REPLY TO THE ATTENTION OF: SR-6J

Mr. Paul Bucholtz
Environmental Quality Analyst
Michigan Department of Environmental Quality
Constitution Hall
525 W. Allegan St., 3rd Floor South
Lansing, MI 48909-7926

RE: Revision 1 Michigan Department of Environmental Quality (MDEQ) Quality Assurance Project Plan (QAPP) for the Allied Paper Incorporated/Portage Creek/Kalamazoo River Superfund Site

Dear Mr. Bucholtz:

The United States Environmental Protection Agency (U.S. EPA) has completed its review of Revision 1 of the MDEQ QAPP for the Allied Paper Incorporated/Portage Creek/Kalamazoo River Superfund Site.

The revised QAPP has adequately addressed U.S. EPA's previous comments and incorporated them into the document. Therefore, U.S. EPA approves Revision 1 of the MDEQ QAPP for the Allied Paper, Incorporated/Portage Creek/Kalamazoo River Superfund Site. U.S. EPA has also enclosed a signed approval page for the QAPP. Please have all of the appropriate officials sign the approval page, and submit a copy of the approval page along with both hard and electronic copies of the final QAPP to U.S. EPA.

If you have any questions regarding this matter, please contact me at (312) 886-0992.

Sincerely,

James A. Saric
Remedial Project Manager
SFD Remedial Response Branch #1

Enclosure

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Michigan Department of Environmental Quality

API/PC/KR Superfund Site
Time Critical Removal Action Monitoring
Quality Assurance Project Plan

January 2008

Draft Report

Quality Assurance Project Plan
TCRA Oversight
API/PC/KR Superfund Site

Revision 1
January 14, 2008
Prepared by: CDM
Prepared for: MDEQ - RRD

Prepared By: _____ Date:_____
CDM Project Scientist

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Project Manager

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CDM Project Manager

Approved By: _____ Date:_____
CDM QA/QC Manager

Approved By: _____ Date:_____
NEA Laboratory Director

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Acronyms and Abbreviations

1,1,1-TCA	1,1,1-trichloroethane
AOC	Administrative Order of Consent
CDM	Camp Dresser & McKee
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program
COC	Chain of Custody
DCA	Dichloroethane
DCE	Dichloroethene
DQO	Data Quality Objective
ECCS	Environmental Chemistry Consulting Services, Inc.
EMSL	Environmental Monitoring Systems Laboratory
FSS	Field Services Section
GC/MS	Gas Chromatography/Mass Spectroscopy
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HS	Hamilton Sundstrand
IAS	Indoor Air Sampling
IEPA	Illinois Environmental Protection Agency
IFB	Invitation for Bid
JP4	Jet Fuel JP4
LCS/LCSD	Laboratory Control Sample/Laboratory Control Sample Duplicate
MDEQ-RRD	Michigan Department of Environmental Quality – Remediation and Redevelopment Division
MDLs	Method Detection Limits
MDNR	Michigan Department of Natural Resources
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NAPL	Non Aqueous Phase Liquid
NELAP	National Environmental Laboratory Accreditation Program
NFG	National Functional Guidelines
OSHA	Occupational Safety and Health Administration
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability

PCE	Tetrachloroethene
PE	Performance Evaluation Sample
PID	Photoionization Detector
PM	Project Manager
ppb	Parts per Billion
ppbv	Parts per Billion by Volume
PQL	Project Quantitation Limit
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
RPD	Relative Percent Difference
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SEDD	Superfund Electronic Data Deliverables
SIM	Selective Ion Monitoring
SOP	Standard Operating Procedure
SOW	Statement of Work
TACO	Tiered Approach to Corrective Action
TCA	Trichoroethane
TCE	Trichloroethene
TCL	Target Compound List
USEPA	United States Environmental Protection Agency
UST	Underground Storage Tank
VC	Vinyl Chloride
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

A.3 Distribution List

The completed Quality Assurance Project Plan (QAPP) and any subsequent revisions will be distributed to the following:

Jim Saric, USEPA Region V, Remedial Project Manager

Mike Ribordy, USEPA Region V

Richard Byvik, USEPA Region V

Paul Bucholtz, MDEQ - RRD

Todd King, CDM

Andrew Santini, CDM

Wendy Dewar, CDM

Arvine Curtis, MDEQ - RRD

William Kotas, Northeast Analytical, Inc.

A.4 Project/Task Organization

CDM/MDEQ has overall responsibility for oversight of the TCRA at the Plainwell impoundment and will conduct and oversee the field investigations and prepare any Technical Memorandum and associated documentation. CDM will also provide quality assurance/quality control (QA/QC) for all deliverables and provide for their issuance.

A.4.A Management Responsibilities

MDEQ-RRD Project Manager, Paul Bucholtz is responsible for overall management and coordination of technical and fiscal aspects of the oversight of the TCRA. Mr. Bucholtz will be the MDEQ contact with the USEPA Region V Project Manager.

The USEPA Region V Project Manager, Jim Saric, and On-Scene Coordinator, Mike Ribordy, are responsible for federal oversight for the TCRA. Mr. Ribordy is responsible for reviewing and analyzing overall performance with respect to goals and objectives.

CDM Project Manager, Todd King, is responsible for the day-to-day management and coordination of the contractor staff. This duty includes, but is not limited to, ensuring that all contractor and subcontractor staff understand and comply with the QA/QC program. The Project Manager is responsible for the Work Plan and review of data generated from field measurements and activities. The Project Manager will also be responsible for preparing the Technical Memorandum. Mr. King is responsible for the following tasks:

- Reviewing all project deliverables prepared by CDM
- Maintaining communications with the MDEQ-RRD Project Manager regarding project status
- Assuring that plans are implemented according to schedule
- Informing personnel of any special considerations associated with the project
- Reviewing work progress for each task to ensure that budgets and schedules are met

A.4.B QA Responsibilities

The Quality Assurance Section Project Officer for MDEQ-RRD is Arvine Curtis. Mr. Curtis is responsible for the QAPP technical review. Mr. Curtis is available for consultation on various QA/QC issues.

Data validation of the subcontractor laboratories, Northeast Analytical, Inc. (NEA) and the MDEQ Laboratory Services Section (MDEQ lab) data will be completed by CDM staff. Data assessment of the data generated in the field will be completed by CDM.

The USEPA Region V Quality Assurance Reviewer is Richard Byvik. Mr. Byvik is responsible for the QAPP and Sampling and Analysis Plan (SAP) review.

The CDM QA Manager is Wendy Dewar. The QA Manager is responsible for data assessment, data validation of subcontractor laboratory data, and oversight of any internal or external performance and system audits.

Robert Avery and William A. Kotas are the Quality Assurance Officers for the MDEQ lab and NEA, respectively. NEA and MDEQ lab will be conducting soil, sediment, surface water and groundwater analysis. Mr. Avery and Mr. Kotas are responsible for the quality of the data generated by their respective laboratories. They are also responsible for reporting the final data in accordance with the requirements of the contract and project. The laboratory selection for sampling events during this project will be determined by laboratory availability and required sample turnaround times.

Internal performance and system audits may be performed by the laboratories at their discretion. External performance and system audits may be performed by CDM, MDEQ, or USEPA as required.

A.4.C Field Responsibilities

CDM field manager, Andrew Santini will coordinate and manage field activities. The field manager will be responsible for coordinating field activities and personnel, making sure the correct number of samples are collected from the appropriate locations, and shipment and documentation of all sampling activities. The field manager will also be responsible for identifying and documenting nonconformance and implementing corrective action. Additional information about field management may be found in the SAP Section 1.2.

A.4.D Laboratory Responsibilities

Soil, sediment, surface water and groundwater analysis will be conducted by the MDEQ lab in Lansing, Michigan, or the NEA Laboratory in Schenectady, New York .

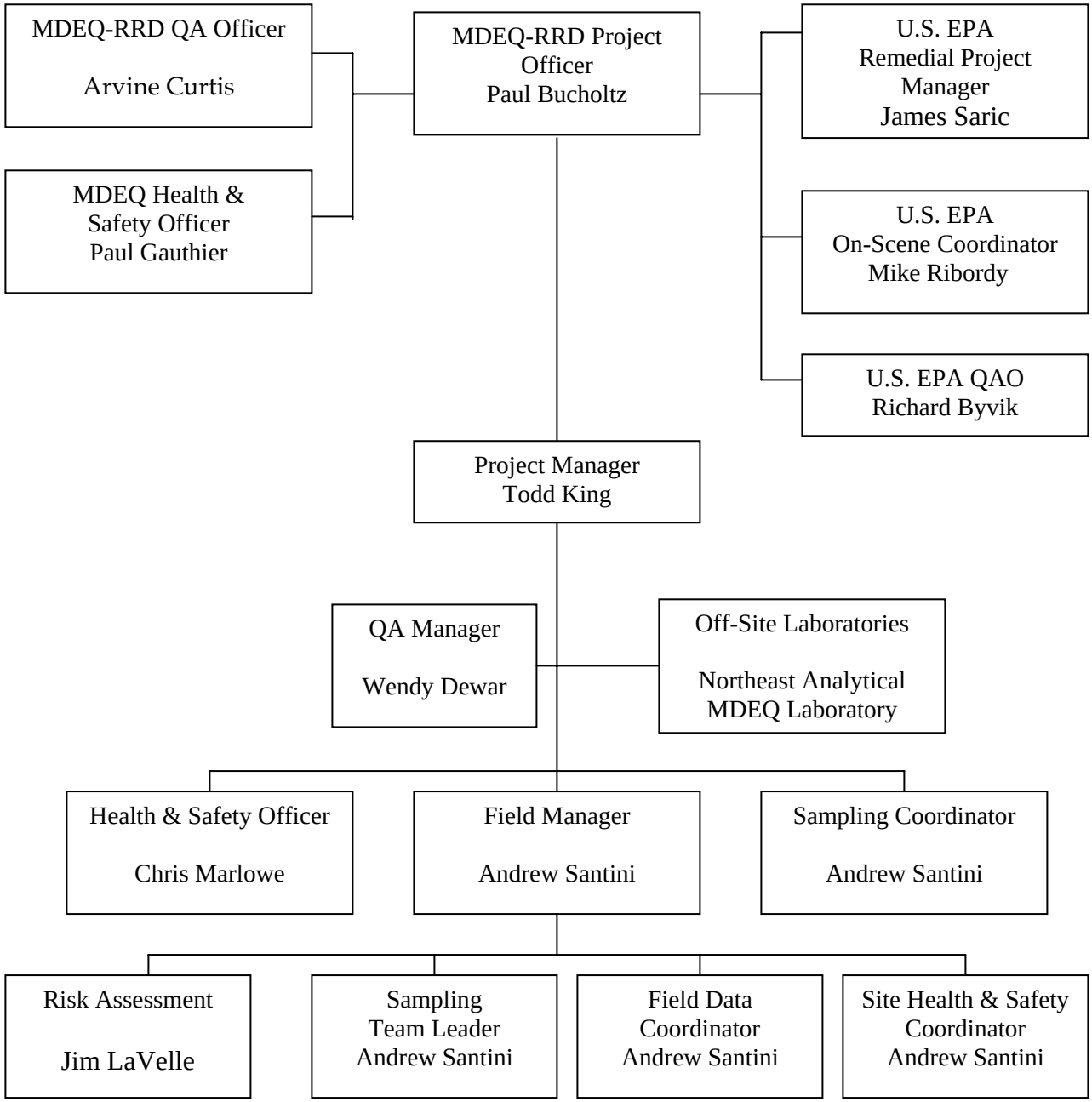
A.4.E Special Training Requirements/Certification

The Health and Safety Officer is responsible for the review of the Health and Safety Plan. The CDM health and safety officer, Chris Marlowe, is responsible for review of the Health and Safety Plan, and ensuring that field personnel and managers have the appropriate 40-hour OSHA Hazwoper, or 8-hour OSHA supervisor training as required.

A.4.F Project Organization Chart

Figure A4-1 shows the project organization chart.

Figure A4-1
Organization Chart
API/PC/KR
Time Critical Removal Action



A.5 Problem Definition for TCRA

EPA and MDEQ have determined that a TCRA is necessary at the API/PC/KR Superfund Site Plainwell Impoundment, and that implementing this action will be a positive step forward in the ongoing investigation and remediation of the API/PC/KR Site. This removal exercise has been described as a "learning exercise" that will be used to determine the utility of similar actions downstream of the Plainwell Impoundment. The engineering design for the TCRA was developed by the Kalamazoo River Study Group (KRSG) and reviewed and approved by the US EPA and MDEQ. The scope of the Design Report is limited specifically to the activities necessary to implement the removal action and is based on the conceptualization that the existing data set provided at the time of report development was sufficient.

Performance monitoring is monitoring designed to describe how well the activities being performed at the site are achieving its objectives. MDEQ field oversight has identified examples that demonstrate where actual conditions in the field differ from the conceptualizations used in the development of the design report. The effective difference between conceptualization and reality can provide for significantly different results than that which was anticipated by following the protocol identified in the Design Report. These conditions create an increased need for performance monitoring at this site by MDEQ/CDM which will allow for the consideration of the significance of the differences identified.

The performance of this TCRA will be evaluated during and after completion by state and federal agencies, the KRSG, and potentially other public and private entities. This performance evaluation will be based on the performance indicator data collected before, during, and after TCRA activities. Collection of this data during and after the removal action will significantly improve efficiencies.

The performance monitoring appropriate for this TCRA include the sampling and analysis of soil, sediment, surface water, and potentially groundwater. These media will be sampled as appropriate based on actual field conditions and MDEQ direction. Performance indicators will be chosen to understand:

- The general nature and extent of the contaminated material left behind.
- The vulnerability and nature of the remaining contaminated material to immediate and eventual introduction/suspension into the river water column.
- The characteristics of contaminated water released into the river water column associated with or a result from the TCRA activities.
- The nature of unanticipated conditions produced as a result of the TCRA activities performed.

The data collected will be used to generally provide for an informed opinion in this learning process for future decision making and for various specific purposes including the following:

- To evaluate the fine scale nature of residual contamination in removal areas
- To be used in conjunction with the long term monitoring data for potentially understanding causes of surface water and fish concentration trends.
- To understand the uncertainty associated with the methods currently used to define "hot spots" for achieving effective removal (i.e., the extent of spatial correlation in interpreted data known as the nugget effect) and guide future decisions about the amount of data needed for defining remedial areas.
- To identify and understand the unintended or unanticipated results of the removal.
- To document the development of various physical features resulting from the removal.
- To evaluate the necessity for more or less field flexibility in future removal work plans (i.e., Design Reports).
- To evaluate and improve the accuracy and utility of the site conceptual model used to date on this site.
- To document the nature of the material not removed and recognize the vulnerability of the material to transport
- To evaluate the relative utility of removal methods in the ongoing investigation and remediation of the Kalamazoo River.

In the current investigation, CDM will collect additional data to evaluate the progress and effectiveness of the TCRA. CDM will collect soil, sediment, surface water and groundwater data to provide the additional information necessary to evaluate the TCRA progress.

A.5.A Background Information (from ARCADIS QAPP)

The United States Environmental Protection Agency (EPA) has determined that the concentrations of polychlorinated biphenyls (PCBs) in the sediments, river bank soils, and floodplain soils of the former Plainwell Impoundment pose an imminent and substantial danger to both human and ecological receptors. EPA has determined that a time-critical removal action (TCRA) is necessary to address the contamination.

The removal action will take place in the former Plainwell Impoundment, which is located in Gun Plain and Otsego Townships, downstream of Plainwell, Michigan. It is roughly bounded on its upstream (or southeastern) end by the Main Street Bridge in Plainwell, and on its downstream (or northwestern) end by the Plainwell Dam. When in operation as a hydroelectric facility, the Plainwell Dam had a head of approximately 13 feet (ft), and it impounded water covering an area of approximately 123 acres. The Michigan Department of Natural Resources

(MDNR) drew down and partially dismantled the dam in the 1970s and 1980s, and as a result, the remaining sill of the dam has a head of approximately 5 ft and impounds a surface area of approximately 44 acres. Currently, the remaining impoundment encompasses approximately 1.9 miles of river, with an average width of 197 ft and an average water depth of 3.7 ft. The channel slope within this reach is approximately 4.6 ft/mile.

Previous Studies of the Former Plainwell Impoundment

The former Plainwell Impoundment has been the focus of an extensive series of investigations since 1993. The nature and extent of PCBs within the impoundment was investigated in a systematic way during the Remedial Investigation/Feasibility Study (RI/FS) field work conducted across the Kalamazoo River Superfund Site in 1993 and 1994. Prior to the RI/FS, the MDNR had also collected analytical and characterization data. In 2000, all available analytical and physical characterization data collected to date were summarized and assessed. In 2001, EPA conducted additional focused sampling of sediments and soils in the former impoundment. A survey of river bank profiles and collection of river bank soil PCB data were performed in 2003. The most recent PCB sediment data were generated during a sampling effort conducted by BBL in 2006. A variety of targeted studies of the impoundment were also conducted in 2005 and 2006 to further characterize Site topography, bank stability/disposition, flow hydrodynamics, equipment accessibility, and habitat quality.

The following briefly summarizes the results of these previous studies, which are organized by channel (i.e., submerged) sediments, bank soils, and floodplain soils (i.e., exposed former sediments).

Sediment

During the 1993 and 1994 RI field work, approximately 125 submerged sediment samples were collected from within the channel of the former Plainwell Impoundment. Total PCB concentrations ranged from non-detect to 139 mg/kg. In 2001, EPA conducted a Phase I sampling program, and collected 53 sediment samples whose total PCB concentrations ranged from non-detect (55% of samples) to 33 mg/kg. During Phase II sampling, EPA collected an additional 160 sediment samples from around sample location SD004, which is approximately 1,500 ft upstream of the Plainwell Dam. The results of this effort showed total PCB concentrations ranging from non-detect (80% of samples) to 4.2 mg/kg; 99% of the samples had total PCB concentrations of less than 1.0 mg/kg. In 2006, BBL collected 222 sediment samples – many of which were judgmentally located to delineate potential "hot spots" – and the total PCB concentrations ranged from non-detect to 220 mg/kg.

Prior to the 2006 sediment sampling, PCBs were not detected in any mid-channel sediment samples at concentrations greater than 1.0 mg/kg – samples with PCB concentrations greater than 1.0 mg/kg from all previous investigations were detected within 40 ft of the shore. However, as a result of BBL's 2006 sediment sampling, three specific mid-channel sediment deposits were identified as containing PCB concentrations greater than 50 mg/kg. For purposes of this TCRA, these three sediment deposits have been designated as Mid- Channel Removal Areas A, B, and C.

Bank Soils

Physical characterization assessments performed during the 1993 and 1994 RI field work revealed that the river banks within the three former impoundments are in many locations susceptible to erosion. This ongoing, uncontrolled erosion is a significant source of PCB loading to the Kalamazoo River, as PCB-containing bank materials slough off the banks to be deposited in the river or transported downstream. The extent and rates of erosion have been estimated based on more than 2 years of measurements developed using erosion pins, which were placed in the banks in 2000. BBL has generated further evidence of erosion through the comparison of 1993 transects and a 2003 survey of 74 bank profiles (placed at 100-ft intervals), each of which was used to demarcate bank slope, top of bank, toe of bank, and bank cross sections. Additional probing and survey data were collected during a series of studies conducted in the former Plainwell Impoundment in 2005 and 2006. Results from the 87 top-of-bank soil samples collected during the 2003 bank characterization study indicate total PCB concentrations in these soils range from 0.20 mg/kg to 120 mg/kg. Thirty samples (34%) contained PCB concentrations less than 5.0 mg/kg. When combined with data from similar locations collected during the 1993/94 RI field work, the arithmetic average total PCB concentration in bank soils was 23 mg/kg, and the median concentration was 13 mg/kg. These combined data also indicate that PCB concentrations are greater in soils near the river channel, and concentrations decrease with distance from the river. These data provide a basis for the delineation of near-shore sediment and bank soil removal areas targeted for the TCRA. Similarly, bank characterization studies, stability analyses, and habitat assessments have provided a basis for optimizing appropriate post-removal restoration conditions such as bank height, bank slope, and habitat characteristics.

Floodplain Soils

The floodplain soils adjacent to the Kalamazoo River and lying within the former Plainwell Impoundment are comprised of approximately 75 acres of former sediments (about 3.8 ft thick) that were exposed after MDNR drew down the impoundment in the early 1970s and dismantled the Plainwell Dam to its sill level in 1987. Today, these upland floodplain soils are covered with vegetation and relatively stable, except for areas along the banks of the river, where the soils are subject to undercutting and erosion, causing them to slough off into the

river. During the RI field work, BBL collected 135 floodplain soil samples that had total PCB concentrations ranging from non-detect to 85 mg/kg. Eighty-seven samples (64%) of the RI soil samples contained PCB concentrations less than 5.0 mg/kg. PCB concentrations were generally found to decrease with depth, and concentrations in subsurface soils tended to decrease with distance from the river. In 2001, EPA collected 147 floodplain soil samples (Phase I), which ranged in total PCB concentration from nondetect (17% of samples) to 84 mg/kg; 76% of samples had concentrations of less than 5.0 mg/kg. EPA conducted Phase II soil sampling in three locations referred to as Grid 1 (218 samples around Sampling Location [SL] 015), Grid 2 (235 samples around SL029), and Grid 6 (159 samples around SL012). Total PCB concentrations in the Phase II samples ranged from non-detect to 158 mg/kg in Grid 1, from non-detect to 45.3 mg/kg in Grid 2, and from non-detect to 65.6 mg/kg in Grid 6. Including all three sample grids, 572 Phase II samples were collected, of which 432 (76%) contained PCB concentrations less than 5.0 mg/kg. In all, 629 of the 854 floodplain soil samples contained PCB concentrations less than 5.0 mg/kg. These data were also used to identify soils with PCB concentrations greater than 50 mg/kg that are subject to removal during the TCRA. The data from these studies served as a basis for the delineation of specific floodplain soil removal areas.

A.6 Project/Task Description and Schedule

An overview of the activities to be performed and the information in this QAPP is included below.

A.6.A Tasks

The following tasks have been established for the Kalamazoo TCRA Monitoring.

A.6.A.1 Task 1

CDM conducted pre-QAPP planning discussions with MDEQ for the TCRA Monitoring. During these discussions, specific information regarding the project schedule, QAPP preparation and review, project goals and objectives, laboratory procurement, and other pertinent project information was discussed.

The purpose of the oversight activities for the TCRA is to confirm that the removal action is being conducted effectively and efficiently based on current site conditions. Data will be gathered in and around removal areas during and after the TCRA activities to allow for evaluation of progress and effectiveness of the removal. This information will be used as input to future actions to be conducted at the API/PC/KR site.

A.6.A.2 Task 2

The data gathering effort for this project will be performed during and after the conduct of the removal action at the Plainwell Dam. The sampling tasks will follow the order that they are occurring during the removal. Soil and sediment samples will be collected during and/or after PRP sampling. Depending on the configuration of the specific removal area, expected concentrations and other field factors, sampling may be conducted in a biased manner (looking for hot spots) or in an unbiased manner consistent with S3TM sampling guidance. Exact sampling locations and numbers can not be determined at this time. As samples are planned and collected this information will be documented and communicated to MDEQ. This data will be used to evaluate extent of any PCB contaminated media left in place and confirm that cleanup has been achieved in designated areas.

A.6.A.3 Task 3

Once it has been determined which of the removal areas will have contamination that remains in place, these areas will be further studied. Areas will be evaluated for insteam stability, bank stability and potential for flooding. Samples will be collected for total PCB and geotechnical parameters.

A.6.A.4 Task 4

CDM will collect surface water and /or groundwater samples as appropriate to evaluate the characteristics of the water being released to the Kalamazoo River during

and after the removal action. Sampling will be concurrent with PRP data and results for total PCB will be compared to PRP data sets. Exact sampling locations and numbers can not be determined at this time as they are dependent on PRP sampling scheme. As samples are planned and collected, this information will be documented and communicated to MDEQ.

A.6.A.5 Task 5

CDM will perform data assessment activities for all data generated during the TCRA Monitoring to ensure that the data is sufficient to support evaluation of current conditions, verification of cleanup and any modifications to the removal action procedures.

Initial data evaluation will be conducted by CDM to determine if the integrity of each data package . If irregularities are identified during the data evaluation process, then data validation will be performed.

If necessary, data validation activities will be performed in accordance with current USEPA CLP guidance. Factors to be considered in data validation include sample holding times, instrument tuning and performance, instrument calibration, blanks, surrogate recoveries, matrix spike/matrix spike duplicate (MS/MSD) analysis, and other QC parameters. The specifications provided in the guidelines will be followed when performing data validation.

Data assessment will be performed upon completion of data evaluation/validation activities. The assessment will be based on all new data and existing data determined to be consistent with the goals of the investigation. Data will be evaluated as it compares to project objectives and summarized into a logical, useable format for data manipulation and interpretation.

Off-site laboratory data will only undergo 100% validation at a Level III validation if irregularities are identified during data evaluation. Further information and details on data validation may be found in Element D1 of this QAPP.

A.6.A.6 Task 6

Quality assurance assessment information may be found in Element C1 of this QAPP.

Performance and systems audits may be conducted for activities conducted by any entity performing services on this project, including off-site laboratories and field team activities.

Performance and systems audits of field activities may be performed periodically by the CDM QA Manager in accordance with CDM audit procedures or the MDEQ PM. Audits will be performed to evaluate sampling activities including sample ID, chain-of-custody, field documentation and proper sampling procedures. The results of the field audits will be reported as part of the Quality Assurance Reports to Management.

A.6.A.7 Task 7

Element D3 of this QAPP includes information about the data usability assessment reconciliation process. DQOs are found in Element A7 of this QAPP. Validated data will be assessed for decision making as specified in the DQOs.

A.6.A.8 Task 8

Element A9 of this QAPP describes all project documents, records and reports that will be compiled and/or generated during the course of this project. Element C2 of this QAPP describes reports to management.

A.6.B Project Schedule

The project schedule is based on the activities to be conducted by ARCADIS.

A.7 Quality Objectives and Criteria for Measurement Data

This section provides internal means for control and review of the project so that environmentally related measurements and data collected are of known and acceptable quality. **Table A7-1** describes the DQOs related to the TCRA Monitoring.

A.7.A Data Quality Objectives

The DQO process is a series of planning steps based on the scientific method that are designed to ensure that the type, quantity, and quality of environmental data used in decision-making are appropriate for the intended purpose. The USEPA has issued guidelines to help data users develop site-specific DQOs. The DQO process is intended to:

- Clarify the study objective;
- Define the most appropriate type of data to collect;
- Determine the most appropriate conditions from which to collect the data; and
- Specify acceptable levels of decision errors that will be used as the basis for establishing the quantity and quality of data needed to support the design.

The goal of the DQO process is to "help assure that data of sufficient quality are obtained to support remedial response decisions, reduce overall costs of data sampling and analysis activities, and accelerate project planning and implementation."

The DQO process specifies project decisions, the data quality required to support those decisions, specific data types needed, data collection requirements, and analytical techniques necessary to generate the specified data quality. The process also ensures that the resources required to generate the data are justified. The DQO process consists of seven steps, and the output from each step influences the choices that will be made later in the process. These steps include:

Step 1: State the problem;

Step 2: Identify the decision;

Step 3: Identify the inputs to the decision;

Step 4: Define the study boundaries;

Step 5: Develop a decision rule;

Step 6: Specify tolerable limits on decision errors; and

Step 7: Optimize the design.

During the first six steps of the process, the planning team develops decision performance criteria (i.e., DQOs) that will be used to develop the data collection design. The final step of the process involves developing the data collection design based on the DQOs. A brief discussion of these steps and their application to this QAPP is provided below.

A.7.A.1 Step 1: State the Problem

Members of the planning team include the following:

- Paul Bucholtz, MDEQ-RRD PM
- Todd King, CDM PM
- Wendy Dewar, CDM QA Manager

The TCRA Monitoring activities have been evaluated through the DQO process. **Table A7-1** identifies the DQOs for the oversight activities.

Due to the collaborative use of the results from sampling in each matrix, the DQO process has not been completed on a matrix basis, but on an activity basis to more effectively meet the intent of the DQO process.

A.7.A.2 Step 2: Identify the Decision

This step identifies what questions the analysis will attempt to resolve and what actions may result. The principal questions for the TCRA Monitoring activities are:

1. In what locations/areas have the TCRA objectives not been met after removal activities, due to site conditions?

2. Will contaminated media not removed pose a significant risk to human health, biota and/or the environment?

3. What is the likelihood that residual contaminated media will migrate and enter the river?

4. What are the characteristics of the water released to the river as a result of the TCRA?

5. Were any unanticipated conditions produced as a result of the TCRA activities?

A.7.A.3 Step 3: Identify the Inputs to the Decision

The purpose of this step is to identify the information that needs to be obtained and the measurements that need to be taken to resolve the decision statements. The inputs required for the TCRA Monitoring decisions include results from the soil, sediment, groundwater and surface water total PCB data as well as visual observations of the removal procedures and results.

A.7.A.4 Step 4: Define the Boundaries of the Study

This step defines the spatial area to which decisions will apply and determines when and where data should be collected. The study boundaries are considered the TCRA removal area and any adjacent areas based on CDM field observations or results. The TCRA removal area is defined by the Administrative Settlement Agreement and Order on Consent for Removal Action. The *Time-Critical Removal Action Design Report for the former Plainwell Impoundment* (Design Report prepared by the PRP consultant ARCADIS BBL) provides a detailed description of the TCRA, including the contract drawings and associated specifications that will be followed during implementation.

A.7.A.5 Step 5: Develop a Decision Rule

The purpose of this step is to define the parameters of interest, specify the action level, and integrate previous DQO outputs into a single statement that describes a logical basis for choosing among alternative actions.

For the soil and sediment sampling in the removal areas, if total PCB concentrations exceed the Project Action Limits (PALs) after removal, the area will be noted as not meeting TCRA objectives and data will be evaluated to determine if additional sampling is required or risk may be evaluated with existing data.

In areas where contamination is left in place due to site conditions, the area will be evaluated to determine flood potential and contaminated media will undergo geotechnical testing and stability evaluation to determine the potential for migration.

Total PCB concentrations in groundwater and surface water that are or have the potential to be released into the Kalamazoo during and after the removal will be compared to PALs. If concentrations exceed PALs during the conduct of the removal action, MDEQ may request that the actions be modified to mitigate the impact of these waters on the River. If concentrations exceed PALs after the conduct of the removal action, MDEQ may request that additional actions be conducted to mitigate the impact of these waters on the River.

A.7.A.6 Step 6: Specify Tolerable Limits on Decision Errors

Decision makers' tolerable limits on decision errors, which are used to establish performance goals for the data collection design, are specified in this step. Decision makers are interested in knowing the true value of the contaminant concentrations. Since analytical data can only estimate these values, decisions that are based on measurement data could be in error (decision error). There are two reasons why the decision maker may not know the true value of the constituent concentration. These are:

(1) Concentrations may vary over time and space. Limited sampling may miss some features of this natural variation because it is usually impossible or impractical to measure every point of a population. *Sampling design error* occurs when the sampling design is unable to capture the complete extent of natural variability that exists in the true state of the environment.

(2) Analytical methods and instruments are never absolutely perfect; hence a measurement can only estimate the true value of an environmental sample. *Measurement error* refers to a combination of random and systematic errors that inevitably arise during the various steps to the measurement process.

The combination of sampling design and measurement error is the total study error. Since it is impossible to completely eliminate total study error, basing decisions on sample concentrations may lead to a decision error. The probability of decision error is controlled by adopting a scientific approach in which the data are used to select between one condition (the null hypothesis) and another (the alternative hypothesis). The null hypothesis is presumed to be true (not rejected) in the absence of evidence to the contrary. For this project the null hypothesis is that the true values of the constituents are within the action levels. The alternative hypothesis is that the true values of the constituents are outside the acceptable criteria.

A false positive or "Type I" decision error refers to the type of error made when the null hypothesis is rejected when it is true, and a false negative or "Type II" decision error refers to the type of error made when the null hypothesis is not rejected when it is false. For this project, a Type I decision error would result in deciding that soil and/or groundwater quality is outside of acceptable criteria when it is within. A Type II decision error would result in deciding that soil and/or groundwater quality was within acceptable criteria when it is outside of acceptable criteria. A Type I decision error may cause spending money to revise the proposed remedy when it is not required whereas, a Type II decision error may cause harm to the environment and/or human health if the proposed remedy does not work as planned due to the presence of these constituents. Therefore, a Type II error is less acceptable than a Type I error.

The chemical concentrations for each matrix will be compared directly to action levels (acceptable criteria). If a value is outside the action level, the media quality is considered unacceptable, and if the result is within the action level, it is assumed acceptable. However, it is acknowledged that due to site conditions it may be impossible to remove all unacceptable media from certain areas of the site. In this case the residual risk associated with these remaining materials will be evaluated.

A.7.A.7 Step 7: Optimize the Design

The data collection program is based on information provided in this QAPP and is presented in Sections 4 and 5 of the SAP.

A.7.B Measurement of Performance Criteria

Every reasonable attempt will be made to obtain a complete set of usable field measurements and analytical data. If a measurement cannot be obtained or is rejected for any reason, the effect of the missing data will be evaluated by CDM. In addition, the SAP provides guidance to ensure that the samples obtained are representative of the media at the site.

The field QA program has been designed in accordance with USEPA's Guidance for the DQO Process, and the USEPA Region V Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan, Revision 0, June 2000.

Precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters are indicators of data quality. PARCC goals are established for the site characterization to aid in assessing data quality. The following paragraphs define these PARCC parameters in conjunction with this project.

Precision. The precision of a measurement is an expression of mutual agreement among individual measurements of the same property taken under prescribed similar conditions. Precision is quantitative and most often expressed in terms of relative percent difference (RPD).

Precision of the laboratory analysis can be assessed by comparing the analytical results and the laboratory duplicate results. The RPD can be calculated for each pair of duplicate analyses using the following equation:

RPD = *S - D*/(S + D)/2 x 100

where:

S = First sample value (original value); and

D = Second sample value (duplicate value).

Precision of reported results is a function of inherent field-related variability plus laboratory analytical variability, depending on the type of QC samples. Field duplicate samples will be collected to provide a measure of the contribution to overall variability of field-related sources. Acceptable RPD limits for field duplicate measurements will be plus or minus 35 percent (%) for aqueous samples and 50% for solid samples. Field duplicates will be collected at a frequency of 1 per 20 investigative samples. Data will not be rejected based on the agreement between field duplicates. Contribution of laboratory-related sources to overall variability will be measured through various laboratory QC samples.

Accuracy. Accuracy is the degree of agreement of a measurement with an accepted reference or true value and is a measure of the bias in a system. Accuracy is quantitative and usually expressed as the percent recovery (%R) of a sample result. The %R is calculated as follows:

$$\%R = SSR - SR / SA \times 100$$

where:

SSR = Spiked Sample Result

SR = Sample Result

SA = Spike Added

Ideally, it is desirable that the reported concentration equals the actual concentration present in the sample. Analytical data can be evaluated for accuracy using matrix spike (MS) samples and laboratory control samples (LCS)s. These QC samples will be analyzed at a rate of 1 per 20 investigative samples.

Representativeness. Representativeness expresses the degree to which sample data represent:

- The characteristic being measured
- Parameter variations at a sampling point, and/or
- An environmental condition

Representativeness is a qualitative parameter that is most concerned with the proper design of the sample plan and sampling procedures and the absence of sample contamination. Acceptable representativeness will be achieved through (1) careful, informed selection of sampling sites, (2) selection of testing parameters and methods that adequately define and characterize the extent of possible contamination and meet the required parameter reporting limits, (3) proper collecting and handling of samples to avoid interferences and prevent contamination and loss, and (4) collection of a

sufficient number of samples to allow characterization. Representativeness is a consideration that will be employed during all sample location and collection efforts. The representativeness can be assessed qualitatively by reviewing the procedures and design of the sampling event, by the establishment and consistent use of SOPs, and quantitatively by reviewing the laboratory blank samples. If an analyte is detected in a laboratory blank, any associated positive result less than five times may be considered undetected.

Completeness. Completeness is a measure of the amount of usable data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions. Usability will be determined by evaluation of the PARCC parameters excluding completeness. Those data that are evaluated and not rejected are usable. Completeness will be calculated following data evaluation. A completeness goal of 95% is projected for the data set collected for the activities at the Site. Completeness will be calculated for the combined data from all sampling events performed during the activities at the Site; completeness will also be calculated for the screening activity and for the indoor air sampling activity. If the completeness goal of 90% is not met, additional sampling may be necessary to adequately achieve project objectives. Completeness is calculated using the following equation:

% Completeness = (DO/DP) x 100

where:

DO = Data Obtained and usable.

DP = Data Planned to be obtained.

Comparability. Comparability is a qualitative parameter. Consistency in the acquisition, handling, and analysis of samples is necessary for comparison of results. Data developed under this investigation will be collected and analyzed using EPA-approved analytical methods and QC measures to ensure comparability of results with other analyses performed in a similar manner. **Table A7-2** identifies the number of field duplicates for each sample matrix.

Sensitivity. Sensitivity, although not a PARCC parameter, will be evaluated for this project. The achievement of MDLs depends on instrument sensitivity and matrix effects. Therefore, it is important for the laboratory to monitor the sensitivity of data-gathering instruments to ensure the data quality through constant instrument performance. Instrument sensitivity will be monitored by the laboratory through the analysis of preparation blanks. CDM will evaluate sensitivity during the entire project by ensuring that reporting limits are below or approach acceptable criteria. Sensitivity criteria are specified in **Table A7-3**.

Table A7-1
DQO for Screening for TCRA Monitoring

Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7
STATE THE PROBLEM	IDENTIFY THE DECISIONS	IDENTIFY INPUTS TO THE DECISIONS	DEFINE STUDY BOUNDARIES	DEVELOP DECISION RULES	SPECIFY LIMITS ON DECISION ERRORS	OPTIMIZE SAMPLING DESIGN
Recent field activities have identified a number of areas where actual field conditions differ from the conceptual site model used to develop the removal design. Timely collection of field data is needed before, during and after TCRA activities to allow for decisions about effectiveness and progress of the removal process. It will be important to identify areas where actual site conditions differ from conceptualized site conditions and consideration of the significance of those differences.	a. What are the concentrations of contaminated media left in place? b. What is the extent of the contaminated media left in place? c. What is the likelihood of contaminated media to be transported to and/or re-suspended into the water column? d. What are the characteristics of the water released to the river associated with or as a result of the TCRA? e. Were any unanticipated conditions produced as a result of the TCRA activities?	a. Soil and sediment sampling for PCBs in and around removal areas. b. Soil and sediment sampling for PCBs in and around removal areas. c. Soil and sediment sampling for PCBs, geotechnical parameters, and flood plain evaluation. d. Groundwater and/or surface water sampling for PCBs in and around removal areas before, during, and after TCRA. e. Visual observations and analysis of the data described above.	a. The TCRA area as defined by the Administrative Settlement Agreement and Order on Consent for Removal Action and additional adjacent areas as determined during field activities.	a. and b. If PCB levels exceed PALs then area will be noted as not meeting cleanup criteria and data will be reviewed to determine if additional evaluations will be needed. c. If contaminated media is likely to migrate or be re-suspended into the river, additional action will be required to mitigate migration and/or erosion. d. If water released to river during TCRA shows contaminants above criteria, removal actions will needed to be modified. e. If unanticipated conditions are produced, site maps and conceptual model will be revised.	Professional judgment will be used to determine practical significance of the test results.	a. Collect biased and unbiased soil and sediment samples from removal areas during and/or after PRPs sampling using S3TM as guidance b. Collect biased and unbiased soil and sediment samples from removal areas during and/or after PRPs sampling using S3TM as guidance c. Evaluate instream, bank stability, and floodplain areas where contamination will remain and collect geotechnical and PCB samples. d. Collect surface water and/or groundwater samples for PCBs in and around removal areas before, during, and after TCRA e. Visual observation and review of CDM and PRP data for TRCA to identify unanticipated conditions.

Table A7-2
Summary of Sampling and Analysis Program

				QC Samples		
Sample Matrix	**Field Parameters**	**Laboratory Parameters [1,2]**	**Investigation Samples[3]**	**Field Dups[4]**	**Field Blanks [5]**	**Matrix Total [3]**
Soil and Sediment Sampling	- Dust Monitor MIE PDR-1000	PCBs	Unbiased determined by S3TM and/or 20 biased per area depending on size	1 in 20	1 in 20	No total Calculated
Surface Water Sampling	- Water Quality (YSI 556 or equivalent) for pH, conductivity, temperature, turbidity, dissolved oxygen, redox potential	PCBs	Transect before, during, after removal activities within and surrounding a removal area	1 in 20	1 in 20	No total Calculated
Groundwater Sampling	- Water Quality (YSI 556 or equivalent) for pH, conductivity, temperature, turbidity, dissolved oxygen, redox potential	PCBs	100% splits until agreement with PRP data	1 in 20	1 in 20	No total Calculated

1.Soil and Sediment - EPA SW-846 8082

2. Surface water – modified congener method 8082; groundwater – low level EPA SW-246 8082
3. MS/MSDs will be collected at frequency of 1 per group of 20 or fewer investigative samples at double sample volume for soil, sediment, surface water and groundwater samples. The number of MS/MSDs to be collected is not included in the matrix total.

4. Soil, sediment, surface water, and groundwater field duplicates will be collected 1 per group of 20 or fewer investigative samples.
5. Field blanks for soil and sediment samples (rinsate blanks if using nondisposable equipment such as hand auger) and for surface water and groundwater samples (field blanks) will be collected at a frequency of one per 20.

Table A7-3 Contaminants of Concern in TCRA Samples
API/PC/KR TCRA Monitoring

Analyte	CAS Number	Project Action Limit (ppb)	NEA Project Quantitation Limits (ppb) (2)	MDEQ Project Quantitation Limits (ppb)	NEA Achievable Laboratory MDLs (ppb)	MDEQ Achievable Laboratory MDLs (ppb)
Total PCB TCRA Oversight Monitoring						
Total PCB -Mid-Channel in-stream sediment	1336-36-3	1000 (3)	50 (4)	100 (7)	15	35
Total PCB-In-stream sediments 40 ft from bank	1336-36-3	1000 (3)	50 (4)	100 (7)	15	35
Total PCB-Soil Floodplain/Residential	1336-36-3	4000 (3)	50 (4)	100 (7)	15	35
Total PCB-Soils-Other Flood plain	1336-36-3	5000 (3)	50 (4)	100 (7)	15	35
Total PCB-Surface Water	1336-36-3	0.2 (1)	0.001 (5)	0.025 (8)	0.0002	NA
Total PCB-Groundwater	1336-36-3	0.2 (1)	0.025 (6)	0.025 (8)	0.011	NA

Notes:

(1) Project Action Limit (PAL) based on lowest of MDEQ Part 201 Generic Cleanup Criteria

(2) NEA Laboratory will be requested to report all detections between the MDL and the PQL and flag them as estimated (J). Analytical Method and Achievable Laboaratoy MDLs are as per the current NEA SOPs

(3) Project Action Limit based on Cleanup Standards for the TCRA in the Enforcement Action Memorandum

(4) EPA SW-846 8082-NEA SOP 148

(5) Modified congener method 8082-NEA SOP 133

(6) Low Level water EPA SW-846 8082-NEA SOP 148

(7) EPA SW-846 8082A- MDEQ SOP 502

(8) EPA SW-846-8082A-MDEQ SOP 502-MDEQ will not report any detections below PQL for water

A.8 Special Training Requirements/Certification

Special training required for this study includes health and safety training, as described in the Health and Safety Plan (HASP) provided as Appendix C of the SAP, and maintenance and calibration of field equipment.

A.9 Documentation and Records

Documents that may be generated throughout the course of the TCRA Monitoring may include the following:

- Sample collection records
- QC sample records
- Field analysis records
- Fixed laboratory records
- Data handling records

A.9.A Documents and Records generated for all aspects of the project

A.9.A.1 Sample Collection Records

The procedure used for sample collection records may be found in Sections 3.5 and 3.6 of the SAP.

A.9.A.2 QC Sample Records

The procedure used for QC sample records may be found in Sections 3.5 and 3.6 of the SAP.

A.9.A.3 Field Measurement Records

CDM will conduct field measurements with field monitoring instruments and will follow the procedures specified in the SOPs for those field instruments (included in the SAP). All results will be recorded in project log books and transferred to report format upon completion of the field activities.

A.9.A.4 Fixed Laboratory Records

The fixed or off-site laboratories (NEA and/or MDEQ lab) for soil, sediment, surface water and groundwater analysis will compile all records as required by their current Laboratory Quality Assurance Plans. NEAs plan is dated June 13, 2007 and MDEQ's current Quality Assurance Manual is dated May 5, 2003 . These records will be included in the data packages to be provided to CDM for data evaluation and validation as required.

A.9.A.5 Data Handling Records

The data handling, reduction, lab validation and reporting for NEA are described in Section 9. Data review and Reporting of their Quality Assurance plan. Section 9.0 Data Reduction, Review, Reporting and Records of the MDEQ Quality Assurance

Manual provides their data handling information. Field data generated by CDM will be handled as described in Section 3.5 of the SAP.

A.9.B Data Reporting Package Format and Documentation Control

The data reporting package formats for both NEA and MDEQ is described in general terms in Section 9, of their respective Quality Assurance plans. Because most data packages are project and client specific, an example of the NEA data package format may be found in Appendix A behind the NEA Laboratory Quality Assurance Plan, June 2007.

Documentation control for NEA is described in Section 11.0, of their Quality Assurance Plan, June 2007. Document control for MDEQ is found in section 9.4 of their Quality Assurance Manual, May 5, 2003.

A.9.C Data Reporting Package Archiving and Retrieval

CDM's local administrative staff in Detroit has the responsibility for maintenance of the document control system. This system includes a document inventory procedure and a filing system. Project personnel are responsible for project documents in their possession while working on a particular task. Field logbook(s) will be filed as part of the document control procedure. All data packages generated for this project will be maintained by CDM in secure project files for a minimum of 10 years. At that time, the files will be returned to the MDEQ for archiving. Documentation describing changes to approved plans, if they occur, will be included in the document control system.

B Data Generation and Acquisition

B.1 Sampling Process Design

The general goals of the TCRA Oversight sampling are to evaluate the effectiveness and progress of the TCRA being conducted at the Plainwell Dam based on current field conditions. Additionally during the conduct of the work areas where actual field conditions differ from those used for removal design will be identified. The sampling process was designed to address these goals.

B.1.A Project Schedule

The project schedule is based on the activities of ARCADIS.

B.1.B Sampling Design Rationale

The sampling design rational for the TCRA Monitoring activities are described in Section 4.0 of the SAP.

B.2 Sampling Methods Requirements

Sampling methods, sample containers, and overall field management are described below.

B.2.A Sampling SOPs

Sampling SOPs may be found in Sections 3, 5, and 6 of the SAP.

Equipment required for the field investigation for sampling, health and safety, documentation, and decontamination is presented in Sections 3.1, 3.5 and Section 5 of the SAP.

B.2.B Cleaning and Decontamination of Equipment/Sample Containers

The sample containers for soil and groundwater will be supplied by the laboratory performing the specific analysis, NEA or MDEQ . NEA and MDEQ maintain copies of the manufacturer certifications for each container lot.

Field equipment cleaning and decontamination procedures may be found in Section 6 of the SAP.

B.2.C Field Equipment Maintenance, Testing and Inspection Requirements

Field equipment will be inspected and maintained in accordance with the preventive maintenance schedule shown on **Table B6-1** of the QAPP. **Table B6-1** also lists spare parts that will be kept for each piece of field equipment. Equipment inspection and maintenance will be documented in the field logbook.

The sampling team leader or field manager will order replacements for defective equipment from the CDM field equipment office. Replacement equipment will be shipped to the site via an overnight courier. If spare parts are used in the field, replacement parts will be ordered from the CDM equipment office and shipped in the same manner (common parts such as batteries may be obtained from a local store).

B.2.D Inspection and Acceptance Requirements for Supplies/Sample Containers

Sections 3 and 6 of the SAP include information on required cleaning, decontamination, and inspection for sample containers and supplies. Criteria in these sections will be reviewed upon receipt of supplies and sample containers to ensure compliance.

B.3 Sample Handling and Custody Requirements

Sample handling and custody requirements for samples and QC samples are described below.

B.3.A Sample Handling

The sample identification and labeling system is described in Section 3.5.2 of the SAP. A description of the sampling methods for soil, sediment, surface water and groundwater are found in SAP Sections 5.1, 5.2 and 5.3, respectively.

Section 4.0 of NEA's Quality Assurance Plan describes laboratory sample handling and is included in **Appendix B**. Section 5.0 of MDEQ's Quality Assurance Manual describes laboratory sample handling and is also included in **Appendix B**.

Sample packaging and shipment is described in Section 3.3 of the SAP. Table 3-1 of the SAP includes information on sample container, volume, preservation, and holding time.

B.3.B Sample Custody

COC procedures and sample shipment will follow the requirements stated in the Sampler's Guide to the CLP and in the USEPA Region V Instructions on the Preparation of a Superfund Division QAPP as described in Element B3. The COC record is employed as a physical evidence of sample custody and control. This record system provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. COC record is employed as physical evidence of sample custody and control. A completed COC record is required to accompany each shipment of samples.

Responsible field personnel for maintaining the COC are the field manager, and the sampling team leader. Responsible laboratory personnel may be found in the laboratory organizational charts. The organizational chart for NEA is found in Appendix B of NEA's Quality Assurance Plan. The organizational chart for MDEQ is provided in figure 2-1 of their Quality Assurance Manual, May 5, 2003.

The NEA Quality Assurance Plan, Section 4.0 (**Appendix B)**, provides details on sample receiving and COC procedures. The MDEQ Quality Assurance Manual (Appendix B) provides details on sample receiving and COC procedures in Sections 5.3 and 5.5.

B.4 Analytical Methods Requirements

Samples collected during this project will be analyzed in accordance with standard USEPA and/or project specific analytical procedures that have been developed from USEPA methods. The purpose of using standard procedures is to provide analytical data of known quality and consistency. Analytical laboratories will adhere to QC requirements as established by USEPA methods.

Laboratory information may be found in Element A.4.D of this QAPP. Following is a description of analytical methods by media type. Analyte groups of interest are summarized in **Table A7-3**. QC samples to be collected are described in the SAP, Section 7.

Soil and Sediment Samples

Samples collected from the soils and sediments in and around the removal areas will be analyzed by an off-site lab, (NEA or MDEQ), for PCB Aroclors reported as total PCBs as provided in **Table A7-3**. NEA's SOPs NE143_03.SOP and NE148_05.DOC describe sample preparation, clean-up, analysis, calibration, standard preparation for solid samples and are included in **Appendix B.** MDEQ's SOPs 400, 401 and 502 describe sample preparation, clean-up, analysis, calibration, standard preparation for solid samples and are included in **Appendix B.**

Surface Water Samples

Surface water samples will be collected during and after the removal action activities. If a lower reporting limit is requested by MDEQ, these samples will be analyzed for PCB congeners reported as total PCB by NEA as provided in **Table A7-3**. NEAs SOPs NE142_06.SOP and NE133_02.SOP describe sample preparation, clean-up, analysis, calibration, standard preparation for aqueous samples and are provided in **Appendix B**. For samples not requiring a lower reporting limit, MDEQ laboratory may be used for surface water sample analysis of PCB Aroclors reported as total PCB. MDEQ's SOPs 403, 405 and 502 describe sample preparation, clean-up, analysis, calibration, standard preparation for aqueous samples and are provided in **Appendix B**.

Groundwater Samples

All groundwater samples collected during the TCRA will be analyzed for low level PCB Aroclors reported as total PCB as provided in **Table A7-3**. NEAs SOPs NE118_06._06.SOP and NE148_05.DOC describe sample preparation, clean-up, analysis, calibration, standard preparation for aqueous samples and are provided in **Appendix B**. MDEQ'ss SOPs 403, 405 and 502 describe sample preparation, clean-up, analysis, calibration, standard preparation for aqueous samples and are provided in **Appendix B**.

B.5 Quality Control Requirements

Field, laboratory, and internal office QC are discussed below.

B.5.A Field Sampling Quality Control

Field sampling QC procedures are described in Section 7 of the SAP. **Table A7-2** lists QC samples that will be collected for each matrix.

QC procedures for field measurements are limited to checking the reproducibility of the measurement by obtaining multiple readings and/or by calibrating the instruments (where appropriate). When any field instrument fails the QC checks for calibration it will be recalibrated, repaired, or replaced, whichever is necessary. QC of field sampling will involve collecting field duplicates and blanks in accordance with the applicable procedures described in the SAP.

Measurement data will be generated in many field activities that are incidental to collecting samples for analytical testing or unrelated to sampling. These activities include, but are not limited to, the following:

Documenting time and weather conditions;
Locating and determining the evaluation of sampling stations;
Determining pH and temperature of water supply; and
Determining depths in a borehole or monitoring well.

The general QC objective for such measurement data is to obtain reproducible and comparable measurements to a degree of accuracy consistent with the intended use of the data through the documented use of standardized procedures. The procedures for performing these activities and the standardized formats for documenting them are presented in the SAP.

B.5.B Analytical Quality Control Checks

NEA Laboratory and MDEQ Laboratory will follow all laboratory QC checks as specified in their SOPs, which include laboratory duplicates, MS and matrix spike duplicate (MSD), LCS, and/or preparation blanks. Analytical QC checks for NEA may be found in the specific methods and Section 10.2 through 10.3 of their Quality Assurance Plan, June 2007, located in **Appendix B**. Analytical QC checks for MDEQ lab may be found in the specific methods and Section 8.1 of their Quality Assurance Manual, May 5, 2003, located in **Appendix B**.

B.6 Instrument/Equipment Testing, Inspection, and Maintenance Requirements

B.6.A Field Instrument Maintenance

The field equipment to be used for this project includes a dust monitor, field pH/conductivity/temperature meter, and turbidimeter. **Table B6-1** shows the Preventive Maintenance schedule for the equipment to be used. The field manager will be responsible for implementing these procedures and documenting the procedures carried out in the field logbook**.**

B.6.B Laboratory Instrument Maintenance

NEA's preventive maintenance procedures are described in Section 5.5.2 of the laboratory's Quality Assurance Plan, June 2007 (refer to **Appendix B**). MDEQ's preventive maintenance procedures are described in Section 11.4 of the laboratory's Quality Assurance Manual, May 5, 2003 (refer to **Appendix B).**

Table B6-1
Preventative Maintenance

INSTRUMENTS	MAINTENANCE PROCEDURES/SCHEDULE	SPARE PARTS IN
Dust Monitor MIE PDR-1000	1. Inspect at beginning of each day and as necessary during use. 2. Check battery, and recharge whe low. 3. Return unit for cleaning/obtain replacement unit as needed 4. Re-calibrate if erratic readings or alteration to unit is noted.	Battery Charger Zeroing Kit
Multi-Parameter Meter YSI 556 (or equivalent)	1. Inspect at beginning of each day and as necessary during use. 2. Check battery, and replace if low. 3. Decontaminate probe at beginning of day and after each use. 4. Return unit for cleaning/obtain replacement unit as needed. 5. Re-calibrate if erratic readings or alteration to unit is noted.	pH/Conductivity Calibration Reagents Spare Batteries
Turbidimeter HACH 2100P	1. Inspect unit at beginning of each day 2. Check battery, and replace if low. 3. Ensure that sample vials are free of dust, dirt, and water spots 4. Measure turbidity of 20 NTU standard daily to ensure calibration is maintained. 5. Return unit/obtain replacement unit if malfunctioning	20, 100, and 800-NTU Turbidity Standards Spare Batteries Kimwipes or soft cloth

B.7 Instrument Calibration and Frequency

B.7.A. Field Instrument Calibration

Field instruments that require calibration include the dust monitor (MIE PDR-1000 Model), pH/Conductivity/Temperature/DO/ORP multiparameter meter (YSI 556), and the turbidimeter (HACH 2100P). These instruments will be calibrated before shipment by the CDM Field Equipment Center in Cambridge, Massachusetts. Due to the short duration of this project, re-calibration of the field instruments is not expected to be needed. Calibration information will be shipped along with the field instrument. This information will be entered into the field book on the first day of sample collection. If erratic readings are noted from a field instrument, or if the instrument appears to be damaged or altered during daily inspections, the instrument will be calibrated in accordance with the manufacturer's instructions. Copies of these manufacturer's instructions can be found in **Appendix C** of the SAP.

The sensitivity of each Field Instrument is as follows:

- Multiparameter Meter (pH probe)- Range 0-14 units, Resolution 0.01 units, Accuracy ±0.01 units.
- Multiparameter Meter (Temperature probe)- Range –5 to 45 °C, Resolution 0.01 °C, Accuracy ±0.15 °C.
- Multiparameter Meter (Conductivity probe)- Range 0 to 100 mS/cm, Resolution 0.001 mS/cm (low range) to 0.1 mS/cm (high range), Accuracy ±0.5% of reading +0.001 mS/cm.
- Multiparameter Meter (Dissolved oxygen probe)- Range 0 to 50 mg/L, Resolution 0.01 mg/L, Accuracy 0 to 20 mg/L, ± 2% of the reading or ±0.2 mg/L, whichever is greater, 20 to 50 mg/L, ± 6% of the reading.
- Multiparameter Meter (Oxidation Reduction Potential probe)- Range -999 to +999 mV, Resolution 0.1 mV, Accuracy ±20 mV.
- Turbidimeter- Range 0-1000 NTU, Resolution 0.01 NTU (0-9.99 NTU range) , 0.1 NTU (10-99.9 NTU range), 1 NTU(100-999.9 NTU range), Accuracy ±2% of reading plus stray light.

The sensitivity required for this project is not greater than that specified for each instrument.

B.7.B Laboratory Instrument Calibration

NEA's instrument calibration procedures are described in Section 6.4 of the laboratory's Quality Assurance Plan, June 2007 and the individual methods (refer to

Appendix B). MDEQ's instrument calibration procedures are described in Section 6.5 of the laboratory's Quality Assurance Manual, May 5, 2003 and the individual methods (refer to **Appendix B**).

B.8 Inspection/Acceptance Requirements for Supplies and Consumables

Prior to acceptance, all supplies and consumables will be inspected by the field manager and/or sampling team leader to confirm that they are in satisfactory condition and free of defects.

B.9 Data Acquisition Requirements

Nondirect measurement data include information from a preliminary assessment and previous sampling events. The acceptance criteria for such data include a review by someone other than the author. Any measurement data included in information from the above sources (i.e., previous sampling events) will determine further action at the site only to the extent that those data can be verified by project staff.

Due to the size of this site and the number of entities that have performed work at the site previously, there is a large quantity of existing data available. Previous data that may be used for this project include soil and sediment characteristics information, historical groundwater data, historical surface water data and historical flow and meteorological data. This data may have been gathered by CDM, MDEQ, BBL and/or ARCADIS BBL. These data sets were gathered under approved QAPPs for site characterization under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). All CDM/MDEQ historical soil and groundwater samples were analyzed by NEA Laboratory using the procedures appended to this QAPP .

B.10 Data Management

Data management information is provided below.

B.10.A Data Recording

Data generated by NEA will be recorded as described in Section 9 of their Quality Assurance Plan in **Appendix B** of this QAPP. Data review at NEA is a multi-step process where data is thoroughly reviewed by the analyst, section supervisor, and/or QA officer and/or Laboratory Director. Ultimately, all data is reviewed and signed off by the laboratory directory or appointed assistant. NEA will be responsible for identifying and correcting errors.

Data generated by MDEQ lab will be recorded as described in Section 9.0 of their Quality Assurance Manual in Appendix B. MDEQ lab data may be entered into the LIMS after peer review or at the completion of the originating analyst review and approval. A draft of data entered into the LIMS is reviewed by a second analyst or supervisor according to laboratory procedures to ensure that the QC is acceptable, calculations are correct, qualifications are noted as required, and to detect transcription errors. Spot checks are performed on computer calculations to verify program validity. Errors detected in the review process are referred to the analyst(s) for corrective action. Once the data is validated, the report is finalized and printed for distribution.

B.10.B Data Validation

Data generated by NEA and MDEQ lab will be evaluated by CDM. The data evaluation tasks include:

- Check package for completeness—ensure all items stated in table of contents are present
- Check traceability of sample from field to lab to data receipt ensuring unique sample numbers are used
- Check condition of samples upon receipt by lab
- Check sample holding times
- Check for appropriate instrument calibration documentation, including initial and continuing calibration checks and whether all calibration criteria were met
- Check for matrix/spike and matrix spike duplicate results and whether they meet criteria
- Check for laboratory control standards and whether they meet criteria
- Check for check standards and whether they meet criteria
- Check each sample for spike recovery and whether they meet criteria
- Check for method blanks and whether they meet criteria

- If possible to identify, check field blanks to see if they meet criteria

Laboratory data will only be validated if irregularities that can not be rectified by the laboratory are identified during the evaluation process. If validation is determined to be necessary then data will be validated as per the USEPA National Functional Guidelines (NFG) for Organic as described in Element D of this QAPP. Field duplicates will be accepted on a 25% RPD criteria. If the 25% RPD criteria is not met, the data will not be rejected, but field sampling and analytical procedures will be reviewed.

B.10.C Data Transformation/Data Reduction

Data reduction procedures for NEA data are provided in Section 8.2 of the NEA Quality Assurance Plan, June 2007 in **Appendix B** of this QAPP. Data reduction procedures for MDEQ lab data are provided in Section 9.1 of the MDEQ Quality Assurance Manual, May 5, 2003 in **Appendix B** of this QAPP.

B.10.D Data Transmittal/Transfer

NEA data will be transferred electronically using ASCII format that is compatible with CDM's database. Additionally, data is transferred in hard copy directly to the QA reviewer for data evaluation. MDEQ lab data will be transferred in hard copy to CDM.

B.10.E Data Analysis

The data from this project will be used as input to the project database for review by project staff and MDEQ. Data will be input directly from the laboratory electronic files for NEA and from hard copy for MDEQ lab data and QC checks will be performed on 100% of the data entry. Any qualification of the data by reviewers or validators will be entered manually.

B.10.F Data Assessment

No statistical computer programs or validation software will be used for this project.

B.10.G Data Tracking

Data generated in the field will be tracked by the CDM field manager and recorded in the field log book. Data generated by the laboratories will be tracked by the CDM field manager at the point of sample scheduling, sample shipment, and data receipt. This information will be kept in a spreadsheet format and maintained with the project files.

B.10.H Data Storage and Retrieval

Electronic data and information files for this project will be maintained on the local area network server in CDM Detroit's office, which is backed up daily on tape backup. All hard copy data will be maintained in CDM project files in the Detroit office. The Project Manager is responsible for storing and maintaining project files. After 10 years, all project files, hard copy and electronic, will be turned over to the MDEQ for archiving.

B.10.I Data Security

Data are maintained in secure files in the CDM's Detroit office. File cabinets are locking cabinets. Both the office and building where the files are located have security personnel at the entrance and all doors are locked after office hours. Electronic data are filed according to project numbers and tape backups are kept in locked storage.

C Assessment/Oversight

C.1 Assessment and Response Actions

Assessment and response actions are described in the following sections.

C.1.A Planned Assessments

Due to the short duration of this project, no external assessments are planned for the laboratories, NEA or MDEQ lab.

Performance and systems audits of field activities will be performed at the start of field activities to minimize impacts of any non-conformances identified. This audit will be conducted by the CDM QA Manager, and/or the CDM PM, in accordance with CDM audit procedures. Audits will be performed to evaluate sampling activities including sample ID, COC, field documentation and proper sampling procedures. The results of the field audits will be reported as part of the Quality Assurance Reports to Management.

For NEA, system audits are performed at scheduled intervals and as warranted by encountered circumstances. System audits may consist of qualitative evaluations of all quality control measurement systems or of selected sub-units. System audits consist of written evaluations of quality control documentation and record keeping, spot checks of procedural compliance with Standard Operating Procedures, review of data generated by quality control samples, review of test documentation and review for compliance with laboratory ethics requirements. System audits are designed to determine whether activities scheduled by the QA Unit are satisfactorily performed. A record of any corrective action taken in response to the audit is maintained.

For NEA, performance audits are conducted at the discretion of management and/or the QA Unit. A performance audit is a quantitative evaluation of the measurement systems for an analytical procedure. Performance audits consist of the submission of samples of known composition under the auspices of the QA Unit with or without the knowledge of the analyst. Single-blind and double-blind performance audits also document whether the analytical method is performed in accordance with the standard operating procedures applicable to the method. The results of the analysis are compared with the known values for conformity with laboratory established acceptance criteria.

Additional information regarding audits for NEA are described in Section 10.4 of their Quality Assurance Plan, June 2007 provided in **Appendix B**.

For the MDEQ lab, annual internal audits are performed to verify that laboratory operations continue to comply with the requirements of the quality system. The quality assurance officer does such audits. Upon completion of the audit, the QA Officer generates an inspection report citing any deficiencies discovered during the

audit. The report is given to the Laboratory Director with a copy to the appropriate Unit Manager and Compliance Officer. It is the ultimate responsibility of the Laboratory Director to ensure that proper corrective action has been implemented on a permanent basis. Where the audit findings cast doubt on the correctness or validity of the laboratory's results, an immediate corrective action is initiated and any client whose work may have been affected is notified. The internal system audits include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc.

At the MDEQ lab at least once per year, laboratory management conducts a review of the quality system to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The review takes account of reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions and other relevant factors.

C.1.B Assessment Findings and Corrective Action Responses

Corrective action reporting for NEA is provided in Section 10.5 of their Quality Assurance Plan, June 2007 provided in **Appendix B**. Corrective action reporting for MDEQ lab is provided in Section 13.0 of their Quality Assurance Manual, May 5, 2003 provided in **Appendix B**.

C.2 Reports to Management

The QA reports will be a part of the regular project reports that CDM submits to the MDEQ PM. In turn these reports may be included reports submitted to USEPA by MDEQ. Field staff will note any quality problems on field data sheets. The CDM PM will inform the project QA Manager upon encountering any quality issues that cannot be immediately corrected. The QA reports will contain but are not limited to:

- project status
- results of performance and systems audits
- data quality assessments
- quality assurance problems with proposed corrective actions
- QAPP amendments
- project meeting minutes
- any unresolved problems
- assessment of data deficiencies
- any other significant QA/QC problems not included above.

D Data Verification/Validation and Usability

Laboratory results will be reviewed for compliance with DQOs. As required by USEPA Region V, all data will be validated based on the project needs. Data validation and evaluation are discussed in the following subsections.

D.1 Data Verification/Validation and Verification Requirements

Data validation is not planned for the data generated by NEA or MDEQ lab unless data evaluation indicates significant non-compliance issues during analysis. The data will be evaluated as described in Section B.10.B and the results will be summarized in the report.

If necessary based on data evaluation, data will be validated with respect to the applicable method compliance, the laboratory specific SOPs and the QAPP requirements. The laboratory SOPs will provide the method specific calibration and QC procedures, the reporting limits and QC acceptance criteria against which the data will be compared. The evaluation criteria provided in the USEPA CLP NFG for Organic Data Review (USEPA, October 1999) will be used only to provide guidance as to how and why qualifications should be applied. **Table D1-1** provides a list of data requirements to be checked during validation. **Table D1-2** provides the checklist to be followed for evaluation and validation of the data.

Data validation consists of examining the sample data package(s) against pre-determined standardized requirements from the laboratory SOPs and QAPP. The validator may examine, as appropriate, the reported results, QC summaries, case narratives, COC information, raw data, LCS/LCSDs, MS/MSDs, initial and continuing instrument calibration, and other reported information to determine the accuracy and completeness of the data package. During this process, the validator will verify that the analytical methodology specified in the QAPP was followed and QC requirements were met. The validator may recalculate selected analytical results to verify the accuracy of the reported information. Analytical results will then be qualified as necessary.

For NEA, data verification includes checking that results have been transferred correctly from laboratory electronic files to the CDM database. For MDEQ lab data verification includes checking that results have been entered correctly from the hard copy into the CDM database. Verification of field results includes checking that results from field log books have been correctly transferred to data reports as well as checking to ensure all deviations from the approved sampling and analysis procedures are well documented.

Table D1-1
Data Review
API/PC/KR Superfund Site
TCRA Monitoring

The data requirements to be checked are listed below:

I. Preservation

II. Gas Chromatograph/Mass Spectrometer (GC/MS) Instrument Performance Check

III. Initial Calibration

IV. Continuing Calibration

V. Blanks

VI. Deuterated Monitoring Compounds (DMCs)

VII. Matrix Spikes/Matrix Spike Duplicates (MS/MSDs)

VIII. Regional Quality Assurance (QA) and Quality Control (QC)

IX. Internal Standards

X. Target Compound Identification

XI. Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

XII. Tentatively Identified Compounds (TICs)

XIII. System Performance

XIV. Overall Assessment of Data

Table D1-2
Data Review Summary
API/PC/KR Superfund Site
TCRA Monitoring

PROJECT NO. _____ SITE _____

LABORATORY _____ NO. OF SAMPLES/MATRIX _____

SDG NO. _____ SOP NO. _____ METHOD REF. _____

REVIEWER NAME _____ COMPLETION DATE _____

LAB MGR: ACTION _____ FYI _____

REVIEW CRITERIA

		FRACTION		
		VOA	BNA	PEST/PCB
1.	PRESERVATION			
2.	GC/MS OR GC/ECD INSTRUMENT PERFORMANCE CHECK			
3.	INITIAL CALIBRATION			
4.	CONTINUING CALIBRATION OR CALIBRATION VERIFICATION			
5.	BLANKS			
6.	DEUTERATED MONITORING COMPOUND SURROGATE SPIKES			
7.	MATRIX SPIKES/MATRIX SPIKE DUPLICATES			
8.	LABORATORY CONTROL SAMPLE			
9.	METHOD QA/QC			
10.	INTERNAL STANDARDS			
11.	FLORISIL CARTRIDE PERFORMANCE CHECK			
12.	TARGET COMPOUND IDENTIFICATION			
13.	COMPOUND QUANTITATION AND REPORTED PQLS			
14.	TENTATIVELY IDENTIFIED COMPOUNDS			
15.	SYSTEM PERFORMANCE			

D.2 Validation and Verification Methods

As previously discussed in Section D.1, data validation will not be conducted for NEA or MDEQ lab data unless data evaluation identifies significant non-conformances that would affect data quality for a specific batch of data. In this event, 100% data validation will be conducted for that batch of data as described in Section D.1. Any data qualifications identified will be added to the data forms in both hard copy and electronic format by the CDM validator and documented in the package narrative. Electronic data will be 100% reviewed to ensure that all qualifiers have been entered and that the electronic data matches the hard copy.

D.3 Usability/Reconciliation with Data Quality Objectives

Once data has been generated, CDM will evaluate the analytical data for the PARCC parameters as stated in Element A.7.B of this QAPP. Data for this project will be used to determine the progress and effectiveness of the removal action being conducted by the PRPs at the Plainwell Dam. Data will be evaluated with respect to usability as it is compared to existing data, PRP collected data and applicable guidelines. Estimated data will be considered usable for all calculations. Sample data will be maintained in the project database. Laboratory QC sample data will be stored in hardcopy (in the project files) and in a separate database.

Appendix A

API/PC/KR Superfund Site
TCRA
Sampling and Analysis Plan

January 2008

Draft Report

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Section 1
Introduction

1.1 Objectives of Sampling Program

This Sampling and Analysis Plan (SAP) describes the sampling activities for performance monitoring during the Time-Critical Removal Action (TCRA) at the Allied Paper, Inc/Portage Creek/Kalamazoo River (API/PC/KR) Superfund Site Plainwell Impoundment. Performance monitoring is monitoring designed to document how well the activities being performed at the site are achieving its objectives. The performance of this TCRA will be evaluated during and after completion by state and federal agencies, the KRSG, as well as other public and private entities. This performance evaluation will be based on the performance indicator data collected before, during, and after TCRA activities.

The performance monitoring appropriate for this TCRA include the collection and analysis of soil, sediment, surface water, and potentially groundwater samples. These media will be sampled as appropriate based on actual field conditions and approved MDEQ guidance. The objectives of the sampling program are to understand:

- The general nature and extent of the contaminated material left behind.
- The vulnerability and nature of the remaining contaminated material to immediate and eventual introduction/suspension into the river water column.
- The characteristics of contaminated water released into the river water column associated with or a result of the TCRA activities.
- The nature of unanticipated conditions produced as a result of the TCRA activities performed.
- The trends in concentrations of different media sampled at the API/PC/KR.
- The relative utility of removal methods in the ongoing investigation and remediation of the Kalamazoo River.

1.2 Sampling Team Responsibilities

Field sampling will be performed by Camp Dresser & McKee Inc. (CDM) and / or the Michigan Department of Environmental Quality (MDEQ). The project organization chart is shown in Figure A4-1 of the MDEQ API/PC/KR Superfund Site Quality Assurance Project Plan (QAPP). Responsibilities of the sampling team are described below.

Field Manager

The Field Manager (FM) will be responsible for assigning the sampling team responsibilities (in conjunction with the MDEQ and CDM Project Managers), as well as overseeing all field activities. The FM will coordinate mobilization and demobilization for the sampling team(s). The FM will be responsible for keeping the MDEQ and CDM Project Managers up to date on all sampling activities. The field manager will be responsible for coordinating field activities and personnel, making sure the correct number of samples are collected from the appropriate locations and shipped to the appropriate labs, and documentation of all sampling activities. The field manager will also be responsible for identifying and documenting nonconformance and implementing corrective action.

Sampling Team Leader

The Sampling Team Leader (STL) will be responsible for the sampling efforts, will assure the availability and maintenance of all sampling equipment and materials, and will maintain an adequate supply of shipping and packing materials. The STL will supervise the completion of all COC records, the proper handling and shipping of the samples collected, be responsible for the accurate completion of field log books, and provide close coordination with the FM, on-site personnel, and off-site laboratory personnel. The STL or FM will be present whenever samples are collected.

Sampling Team Member(s)

The Sampling Team Member(s) (STM) will perform field measurements, collect samples, prepare samples for shipping, and decontaminate sampling equipment as directed by the STL.

Site Health and Safety Coordinator (SHSC)

The SHSC is responsible for daily supervision and documentation of all safety, decontamination, environmental monitoring, and field medical monitoring activities. The SHSC is responsible for assuring that CDM field personnel comply with the provisions of the CDM Health and Safety Plan (HSP). The SHSC has the authority to suspend site work if conditions become unsafe, if HSP requirements are not met, or if he/she determines that an upgraded level of protection may be required. The SHSC is responsible for designating and marking restricted areas during various site activities and for re-designating these areas when it is appropriate to do so.

1.3 Scope of Sampling Activities

The purpose of the oversight activities for the TCRA is to confirm that the removal action is being conducted effectively and efficiently based on current site conditions. Data will be gathered in and around removal areas during and after the TCRA activities to allow for evaluation of progress and effectiveness of the removal. TCRA oversight and sampling will depend on the progress of the PRPs removal action and

their planned sampling schedule. The following field activities will be performed to generally provide for an informed opinion in this learning process for future decision making at the API/PC/KR site.

- Soil and sediment samples may be collected during and/or after PRP excavation and sampling. Depending on the configuration of the specific removal area, expected concentrations and other field factors, sampling may be conducted in a biased manner (looking for hot spots) or in an unbiased manner consistent with S3TM sampling guidance. The exact sampling locations and number of samples to be collected can not be determined at this time, however, these will be determined by CDM field staff in conjunction with the MDEQ PM.

- Surface water and /or groundwater samples will be collected as appropriate to evaluate the characteristics of the water being released to the Kalamazoo River during and after the removal action. Depending on the configuration of the specific removal area, expected concentrations and other field factors, sampling may be conducted in a biased manner (looking for hot spots) or in an unbiased manner. The exact sampling locations and number of samples to be collected can not be determined at this time, however, these will be determined by CDM field staff in conjunction with the MDEQ PM.

Table 1-1 is a summary of the sampling and analysis program. The numbers of samples collected during TCRA oversight activities will depend on the progress of the PRPs removal action and their planned sampling schedule as a result, the actual numbers of samples collected will likely vary somewhat from those given in this SAP, or in the Work Plan and QAPP Addendum.

Table 1-1
Summary of Sampling and Analysis Program

				QC Samples		
Sample Matrix	**Field Parameters**	**Laboratory Parameters [1,2]**	**Investigation Samples[3]**	**Field Dups[4]**	**Field Blanks [5]**	**Matrix Total [3]**
Soil and Sediment Sampling	-	PCBs	Unbiased determined by S3TM and/or biased per area depending on size	1 in 20	1 in 20	No total Calculated
Surface Water Sampling	- Water Quality (YSI 556 or equivalent) for pH, conductivity, temperature, turbidity, dissolved oxygen, redox potential	PCBs	Transect before, during, after removal activities within and surrounding a removal area	1 in 20	1 in 20	No total Calculated
Groundwater Sampling	- Water Quality (YSI 556 or equivalent) for pH, conductivity, temperature, turbidity, dissolved oxygen, redox potential	PCBs	100% splits until agreement with PRP data	1 in 20	1 in 20	No total Calculated

1.Soil and Sediment - EPA SW-846 8082

2. Surface water – modified congener method 8082; groundwater – low level EPA SW-246 8082
3. MS/MSDs will be collected at frequency of 1 per group of 20 or fewer investigative samples at double sample volume for soil, sediment, surface water and groundwater samples. The number of MS/MSDs to be collected is not included in the matrix total.

4. Soil, sediment, surface water, and groundwater field duplicates will be collected 1 per group of 20 or fewer investigative samples.
5. Field blanks for soil and sediment samples (rinsate blanks if using nondisposable equipment such as hand auger) and for surface water and groundwater samples (field blanks) will be collected at a frequency of one per 20.

Section 2
Project Description

2.1 Background Information (from ARCADIS QAPP)

The United States Environmental Protection Agency (EPA) has determined that the concentrations of polychlorinated biphenyls (PCBs) in the sediments, river bank soils, and floodplain soils of the former Plainwell Impoundment pose an imminent and substantial danger to both human and ecological receptors. EPA has determined that a time-critical removal action (TCRA) is necessary to address the contamination.

The removal action will take place in the former Plainwell Impoundment, which is located in Gun Plain and Otsego Townships, downstream of Plainwell, Michigan. It is roughly bounded on its upstream (or southeastern) end by the Main Street Bridge in Plainwell, and on its downstream (or northwestern) end by the Plainwell Dam. When in operation as a hydroelectric facility, the Plainwell Dam had a head of approximately 13 feet (ft), and it impounded water covering an area of approximately 123 acres. The Michigan Department of Natural Resources (MDNR) drew down and partially dismantled the dam in the 1970s and 1980s, and as a result, the remaining sill of the dam has a head of approximately 5 ft and impounds a surface area of approximately 44 acres. Currently, the remaining impoundment encompasses approximately 1.9 miles of river, with an average width of 197 ft and an average water depth of 3.7 ft. The channel slope within this reach is approximately 4.6 ft/mile.

Previous Studies of the Former Plainwell Impoundment

The former Plainwell Impoundment has been the focus of an extensive series of investigations since 1993. The nature and extent of PCBs within the impoundment was investigated in a systematic way during the Remedial Investigation/Feasibility Study (RI/FS) field work conducted across the Kalamazoo River Superfund Site in 1993 and 1994. Prior to the RI/FS, the MDNR had also collected analytical and characterization data. In 2000, all available analytical and physical characterization data collected to date were summarized and assessed. In 2001, EPA conducted additional focused sampling of sediments and soils in the former impoundment. A survey of river bank profiles and collection of river bank soil PCB data were performed in 2003. The most recent PCB sediment data were generated during a sampling effort conducted by BBL in 2006. A variety of targeted studies of the impoundment were also conducted in 2005 and 2006 to further characterize Site topography, bank stability/disposition, flow hydrodynamics, equipment accessibility, and habitat quality.

Section 3
General Sampling Information

Collection, preservation, documentation, and shipping of site-specific samples is included in the following sections. However, the sampling procedures will follow applicable guidelines in the field sampling guidance and are not limited to the information provided in Section 3.

3.1 Sample Containers and Preservation

Soil, sediment, surface water, and groundwater samples will be placed in sample containers provided by either Northeast Analytical Laboratory (NEA) or the MDEQ lab, depending on which lab the samples get sent to. Containers for samples that require preservative will be pre-preserved by the manufacturer. Sampling, handling and shipping of the samples will be performed in accordance with these anticipated concentrations.

A summary of soil, sediment, surface water, and groundwater sample containers, their size and construction material, and sample matrix and holding times is given in **Table 3-1**. Internal laboratory QC requirements for analytical samples are discussed in the QAPP.

The sample containers and preservation, if required, will be as follows:

Table 3-1
Sample Volumes, Containers, and Preservation Techniques

Analysis	Container	Preservative	Maximum Holding Time	Volume of Sample	Matrix
Total PCBs	One 4-ounce glass jar (NEA or MDEQ lab)	Cool to 4°C	Extract within 10 days of receipt; analyzed within 30 days of extraction	Fill completely	Sediment / Soil
Total PCBs	One 1-liter amber glass bottle (NEA) Two 1-liter amber glass bottles (MDEQ lab)	Cool to 4°C	Extract within 5 days of receipt; analyzed within 30 days of extraction	Fill completely	Surface Water / Groundwater

Notes :
(1) At critical sampling locations, two 1-liter amber glass bottles will be sent to NEA in case reanalysis is needed. These critical sampling locations will be determined at the discretion of the MDEQ.

3.1.1 Soil and Sediment Samples

- Soil and sediment samples collected for total PCB analysis will be collected in one 4-ounce glass jar.

- At sample collection points where duplicate samples will be collected, double sample volume will be collected, but labeled and submitted to the designated lab for analysis as separate samples with different sample IDs.

- At sample collection points where MS/MSD samples will be collected, triple sample volume will be collected and labeled with the same sample ID as the original sample, but with a 'MS' or 'MSD' on the label. The COC will designate the sample as an MS/MSD with triple volume.

- All soil and sediment samples will be cooled at 4°C in an iced cooler following individual sample collection.

3.1.2 Surface Water and Groundwater Samples

- Surface water and groundwater samples will be collected in one 1-liter amber glass bottle for NEA and two 1-liter glass amber bottles for the MDEQ lab. At critical sampling locations, two 1-liter amber glass bottles will be sent to NEA in case reanalysis is needed. These critical sampling locations will be determined at the discretion of the MDEQ.

- At sample collection points where duplicate samples will be collected, double sample volume will be collected, but labeled and submitted to the designated lab for analysis as separate samples with different sample IDs.

- At sample collection points where MS/MSD samples will be collected, triple sample volume will be collected and labeled with the same sample ID as the original sample, but with a 'MS' or 'MSD' on the label. The COC will designate the sample as an MS/MSD with triple volume.

- All water samples will be cooled at 4°C in an iced cooler following individual sample collection.

3.2 Sample Holding Times

The sample holding times for sediment, soil, surface water, and groundwater samples are listed on Table 3-1. To expedite sample analysis, the samples will most likely be shipped via an overnight carrier on the day of or the day after sample collection. If sampling occurs on a Friday or over the weekend, samples will be cooled at 4°C in an iced cooler and shipped via an overnight carrier on Monday.

3.3 Sample Packaging and Shipment

Following sampling, the sample bottle exteriors will be wiped free of materials and/or rinsed with potable or distilled water prior to shipment. The FM will help the STL and/or STMs prepare documentation and package sample bottles for shipment according to the following procedures.

- Tighten sample bottle caps securely; mark liquid levels if bottles are partially full.
- Label each individual sample as soon as practical after collection. Sample labels will be checked for completeness and accuracy. After checking, attach clear packing tape over with sample label to protect information.
- Complete COC forms for collected samples in the field, or as soon as practical after collection of each individual sample, using information from the field notebook.
- Place containers in a cooler and maintain at 4°C with cold packs or ice sealed in plastic bags; fill remaining space in cooler with additional packing material.
- Place completed COC forms in a zip-loc bag and tape to inside of cooler lid.
- Close cooler and seal with strapping or packing tape; if cooler has a drain port, seal it with duct tape; place one custody seal across closure at front of cooler and across hinge area at back of cooler, or rear side corner.
- Affix airbill with shipper's and consignee's addresses to top of cooler; place "This End Up" labels appropriately.
- The STL or STMs will notify NEA sample coordinator by phone or email that samples are being shipped. The sample matrices, number of samples, airbill number, and any other pertinent sample information will be communicated to NEA. The shipping status of the cooler(s) will be monitoring using the carrier's online system to ensure the cooler(s) are delivered to NEA. If the tracked cooler has not arrived on time, the STL will contact NEA and / or the shipping company to investigate. MDEQ PM will be kept up-to-date on the status sampling and shipping, and if any problems have occurred in the process.

3.4 Chain-of-Custody Procedures

COCs will be maintained throughout the sample preparation procedure as described in the QAPP.

3.5 Documentation

This section outlines the documentation required for sample collection, handling and shipment to be conducted during the TCRA oversight activities.

3.5.1 Field Log Books

Field log books will provide the means of recording pertinent data collected during the performance of field activities. As such, entries will be described in as much detail as possible so that site personnel can reconstruct a particular situation without reliance on memory.

Field log books will be bound, usually consisting of waterproof paper. CDM and MDEQ will use separate log books to document activities and will be stored in the appropriate storage area when not in use. All CDM field generated data will be retained by CDM in permanent files in CDM's Detroit Office (One Woodward Avenue, Ste. 1500, Detroit, Michigan, 48226). One copy of the field data will be delivered to the MDEQ project manager on request.

The title page of each notebook will contain:

- Person or Organization (CDM or MDEQ);
- Book Number;
- Project Name;
- Start Date; and
- End Date.

Entries into the log book will contain a variety of information. At the beginning of each entry, the date, start time, weather, name of all team members present, level of personal protection being used, and the signature of the person making the entry will be recorded. The names of visitors to the site, field sampling or investigation team personnel and the purpose of their visit will be recorded in the field log book. At the end of each day's activity, the log will be closed with the date and/or and signature of the person making the last entry (log-closed line). The log-closed lines and the following log-open lines will be placed so that no unauthorized entries can be made between entries.

Measurements made and samples collected will be recorded. Entries will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Wherever a sample is collected or a measurement is made, a detailed description of the location of the station, which may include compass and distance measurements, shall be recorded, along with GPS

coordinates if recorded. The number of the photographs taken of the station with a brief description including the direction faced will be noted. All equipment used to make measurements will be identified, along with the date of calibration.

The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume, and number of containers. Sample location identifiers will be assigned prior to sample collection.

3.5.2 Sample Identification System

Sample Number

Efficient and accurate data handling begins with properly labeling the samples in the field. All samples will be assigned a unique sample number. The sample designations will be as follows:

Paa-bbbb-cc-dd

where P=Plainwell Impoundment TRCA samples, aa = sample matrix, bbbb=removal area, station or well number, cc=location within a removal area or station , and dd=sample QA/QC designator (for blanks, duplicates, etc.). If a sample ID other than those identified below is reported, the STL will be notified of the modification and rationale of the deviation. For a further explanation on how a sample number is determined, please see below.

aa = Sample Matrix

This two letter designation identifies the sample based on the matrix. The table below outlines the abbreviations for surface water, groundwater, sediment, and soil samples.

Two Letter Abbreviation	Matrix
SW	Surface Water
GW	Groundwater
SD	Sediment
SL	Soil

bbbb = Removal Area, Station, or Well Number

The intent of the removal area, station or well number is to capture the pre-designated location identifier (which may be greater or less than four characters). Soil, sediment, and surface water samples will usually be collected from within or near a removal area designated by the PRPs. Currently, there removal areas are numbered 1 through 12, with some being identified with an "A" or "B". Groundwater samples will most likely be collected from monitoring that have names designated by the PRPs. The table below outlines some examples of abbreviations for stations.

Two Letter Abbreviation	Station	Examples
RA	Removal Area	-RA1-; -RA12A-
MW	Monitoring Well	-MW1-; -MW12-

cc = Location within a removal area or station

This first numerical digit sequentially identifies each location sampled in 2D within a removal area (note that this may actually be two digits depending on the name of the removal area). For example, the first sediment core collected in a removal area is labeled '1', while the twentieth sediment core would be labeled '20'. The second digit (or third) is a letter identifying a location in the third dimension. For example, a sediment core sectioned into three distinct samples from top to bottom would have A, B, and C designated for the sampled intervals. A value of 0 is given for groundwater samples, unless two samples are collected from the same well on the same day or if groundwater samples are collected in the same well but different depths in the screen.

dd = the type of sample from this station at this transect.

This two digit number designation identifies the sample QA/QC. The values

-01 first or original sample ("MS" or "MSD" after sample if appropriate)
-02 blank sample
-03 duplicate sample
-04 control standard

Sample designations will be recorded in the sample field book, on the COC forms, and on the sample labels affixed to the sample jars.

3.6 Sample Documentation Forms

CDM will follow CLP-like procedures for preparation of sample labels, COC preparation, and sample packaging/shipping procedures for samples, as applicable. COCs will accompany all samples to the laboratory.

A) COC Form (see image below)

1) One form per shipping container will be used.

2) Carrier service will not need to sign form if custody seals remain intact.

3) Will be used for all samples.

NEA COC Form

CHAIN OF CUSTODY RECORD

PAGE _____ OF _____

NORTHEAST ANALYTICAL, INC.
2190 Technology Drive, Schenectady, NY 12308
Telephone (518) 346-4592 Fax (518) 381-6055
www.nealab.com information@nealab.com

LRF # _____
(NEA USE ONLY)

DISPOSAL REQUIREMENTS: (To be filled in by Client)
- ◯ RETURN TO CLIENT
- ◯ DISPOSAL BY NORTHEAST ANALYTICAL
- ◯ ARCHIVAL BY NORTHEAST ANALYTICAL

Additional charges incurred for disposal (if hazardous) or archival. Call for details.

CLIENT (REPORTS TO BE SENT TO):
PROJECT#/PROJECT NAME:
PROJECT MANAGER:
LOCATION (CITY/STATE) ADDRESS:
PHONE:
SAMPLED BY: (Please Print)
REQUIRED TURN AROUND TIME:
SAMPLING FIRM:
NAME OF COURIER (IF USED):
FedEx #
Data Package: ☐ Full ☐ Certificates Only
ELECTRONIC RESULTS ☐ E-MAIL ADDRESS:
FAXED RESULTS ☐ FAX #:

ENTER ANALYSIS AND METHOD NUMBER REQUESTED

PRESERVATIVE CODE:
BOTTLE TYPE:
BOTTLE SIZE:
NUMBER OF CONTAINERS

PRESERVATIVE KEY
0 - NONE
1 - HCL
2 - HNO3
3 - H2SO4
4 - NaOH
5 - Zn. Acetate
6 - MeOH
7 - NaHSO4
8 - Other _____

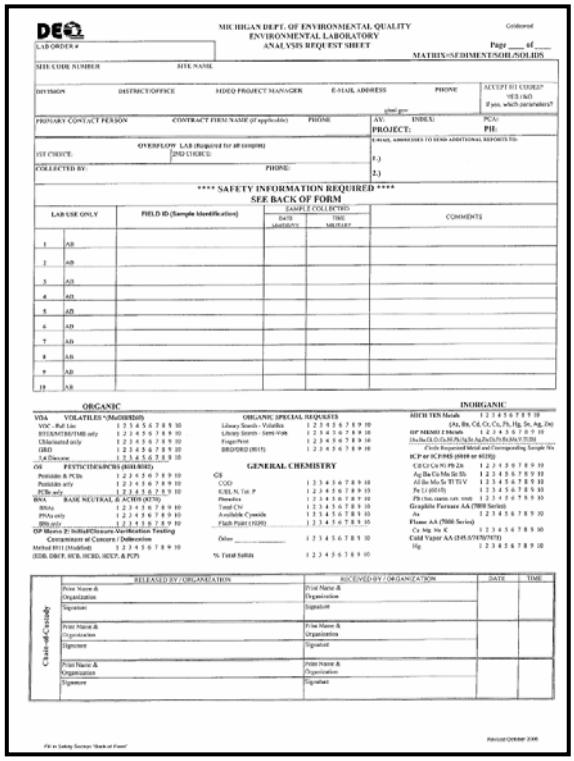
SAMPLE ID	DATE	TIME	MATRIX	GRAB/ COMP	LAB SAMPLE ID (NEA USE ONLY)	REMARKS:

AMBIENT OR CHILLED: TEMP:
COC TAPE: Y N
PROPERLY PRESERVED: Y N
OTHER NOTES:
RECEIVED BROKEN OR LEAKING: Y N
COC DISCREPANCIES: Y N
RECVD W/I HOLDING TIMES: Y N

RELINQUISHED BY	RECEIVED BY	RELINQUISHED BY	RECEIVED BY	RELINQUISHED BY	RECEIVED BY
SIGNATURE	SIGNATURE	SIGNATURE	SIGNATURE	SIGNATURE	SIGNATURE
PRINTED NAME	PRINTED NAME	PRINTED NAME	PRINTED NAME	PRINTED NAME	PRINTED NAME
COMPANY	COMPANY	COMPANY	COMPANY	COMPANY	COMPANY
DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME

S:\ADMIN\COC FORMS.XLS (Revised May 2, 2005)

MDEQ COC – 2 Pages



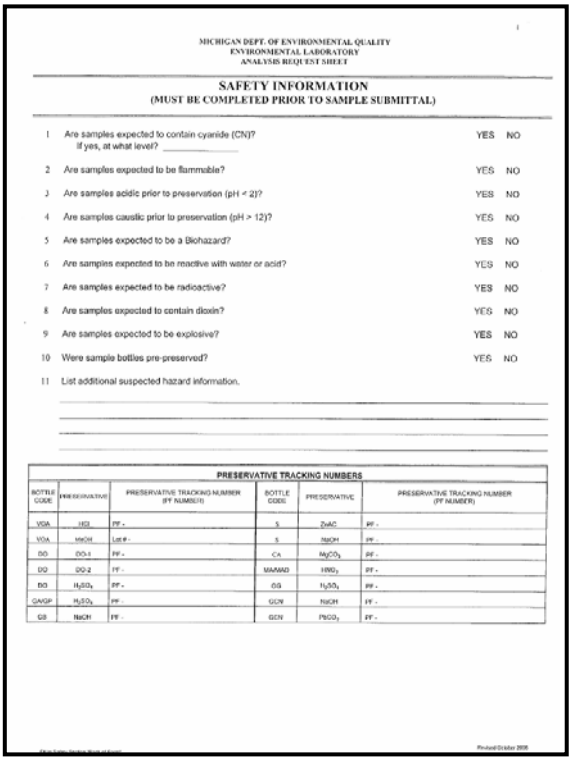
MICHIGAN DEPT. OF ENVIRONMENTAL QUALITY
ENVIRONMENTAL LABORATORY
ANALYSIS REQUEST SHEET

**** SAFETY INFORMATION REQUIRED ****
SEE BACK OF FORM

ORGANIC | ORGANIC SPECIAL REQUESTS | INORGANIC

GENERAL CHEMISTRY

Chain-of-Custody



MICHIGAN DEPT. OF ENVIRONMENTAL QUALITY
ENVIRONMENTAL LABORATORY
ANALYSIS REQUEST SHEET

SAFETY INFORMATION
(MUST BE COMPLETED PRIOR TO SAMPLE SUBMITTAL)

1	Are samples expected to contain cyanide (CN)? If yes, at what level?	YES NO
2	Are samples expected to be flammable?	YES NO
3	Are samples acidic prior to preservation (pH < 2)?	YES NO
4	Are samples caustic prior to preservation (pH > 12)?	YES NO
5	Are samples expected to be a Biohazard?	YES NO
6	Are samples expected to be reactive with water or acid?	YES NO
7	Are samples expected to be radioactive?	YES NO
8	Are samples expected to contain dioxin?	YES NO
9	Are samples expected to be explosive?	YES NO
10	Were sample bottles pre-preserved?	YES NO
11	List additional suspected hazard information.	

PRESERVATIVE TRACKING NUMBERS

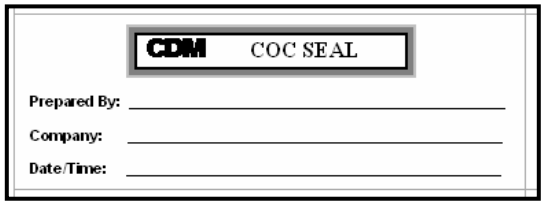
B) COC Seals (see image below)

1) Two seals per shipping container will be used to secure the lid and provide evidence that samples have not been tampered with.

2) Seals will be covered with clear tape.

3) Seals will be used for all sample shipping containers.

4) The STL or FM will sign and date each custody seal.



C) Sample Labels (see image below)

1) A sample container from each sampling location will have a sample label affixed to it with clear packing tape around the container.

2) The sample ID, date, time, analysis required, and preservativewill be recorded on the sample label.

Northeast Analytical – Environmental Lab Services **2190 Technology Drive, Schenectady, NY 12308**
Company Name: MDEQ/CDM
Sample ID:
Sample Location:
Matrix:
Required Analysis:
Date: **Time:**
Sampled By:

The COC will be sealed in a plastic bag that is taped to the inside of the cooler lid. Copies of the COC will be retained for the project files.

Section 4
Sampling Locations and Rationale

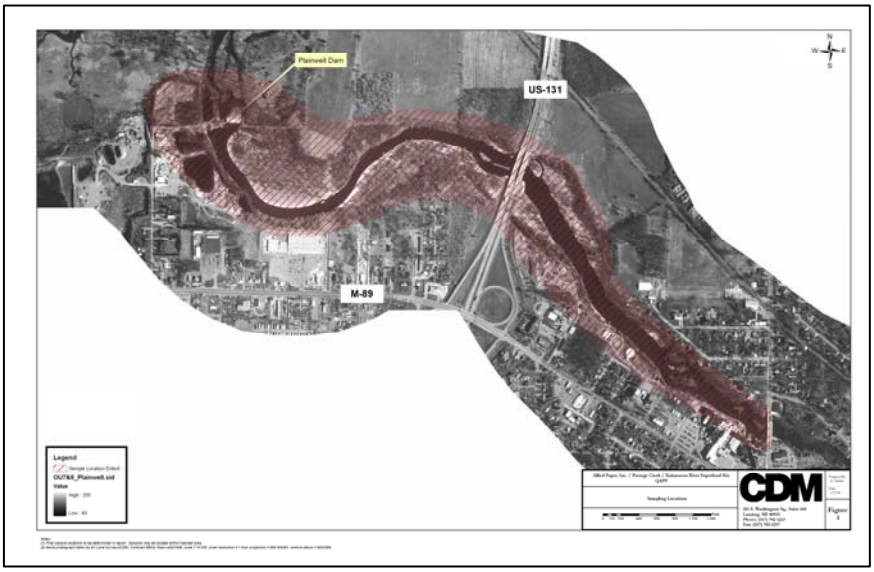
Exact sampling locations within or near a removal area will be chosen in the field, by the sampler, and governed by site conditions at the time of sampling. Where possible, sampling location coordinates will be recorded using a hand held GPS. Additionally, detailed written notes of the sample location (position in the river channel, approximate distance to shore, proximity to local landmarks, etc.) will be recorded.

4.1 Sediment and Soil Sampling

Soil and sediment samples may be collected during and/or after PRP excavation and sampling. Depending on the configuration of the specific removal area, expected concentrations and other field factors, sampling may be conducted in a biased manner (looking for hot spots) or in an unbiased manner consistent with S3TM sampling guidance. The exact sampling locations and number of samples to be collected can not be determined at this time, however, these will be determined by CDM field staff in conjunction with the MDEQ PM and are likely to be contained within the hatched area of the figure below.

4.2 Surface Water and Groundwater Sampling

Surface water and /or groundwater samples will be collected as appropriate to evaluate the characteristics of the water being released to the Kalamazoo River during and after the removal action. Depending on the configuration of the specific removal area, expected concentrations and other field factors, sampling may be conducted in a biased manner (looking for hot spots) or in an unbiased manner. The exact sampling locations and number of samples to be collected can not be determined at this time, however, these will be determined by CDM field staff in conjunction with the MDEQ PM and are likely to be contained within the hatched area of the figure below.



Section 5
Sampling Equipment and Procedures

5.1 Sediment and Soil Sampling

Instructions presented in this Standard Operating Procedure (SOP) are for collecting representative sediment samples from surface water bodies. Sediment can be considered as any material that is submerged/saturated (at least temporarily) or suspended in any surface water body. This includes sludges, lake bottom sediments, perennial and intermittent stream sediments, and marine sediments.

Sampling Methods

Presented below are sampling instructions for the most common tube sampling technique for collecting sediment samples. Prior to sample collection, water body characteristics (size, depth, flow) should be recorded in the field logbook. Sampling should proceed from downstream locations to upstream locations so that disturbance from sampling does not affect sampling quality. In collecting sediment samples from any source, care must be taken to minimize disturbance and sample washing as it is retrieved through the liquid column. Sediment fines may be carried out of the sample during collection if the liquid above is flowing or deep. This may result in collection of a non-representative sample due to the loss of contaminants associated with these fines.

While a sediment sample is usually expected to be a solid matrix, the sampler should not place the sample in the bottle and decant the excess liquid. If the sample is collected properly, any liquid in the bottle is representative of sediment conditions. If the liquid above the sediment collection point is either flowing or greater than 6 inches in depth, a corer or other device that eliminates sample washing must be used to collect the sample to minimize washing the sediment as it is retrieved. It may be necessary to decant standing water from the top of the core. This should be done carefully and prior to transfer to the sample bottle.

Sample Types

The type of sample should be designated when selecting a sampling method. Sediment samples can either be discrete or composite samples. A discrete sample is defined as a single aliquot from of a specific location at a given point in time. Composites are samples composed of two or more specific aliquots (discrete samples) collected from one or several sampling locations and/or different points in time. This type of sample represents an average value and can, in certain instances, be used as an alternative to analyzing a number of individual discrete samples and calculating an average value. It should be noted, however, that compositing can mask the presence of contaminants by diluting isolated concentrations of analytes that may be present in the environmental matrix.

Sampler Selection

The choice of samplers is dictated by sampling objectives (surficial versus subsurface samples) and site constraints. Each sampling technique presents various advantages and disadvantages for its application. For example, sample disturbance, sample volume, chemical and physical reactivity between potential contaminants and sampling tool materials, and ease of decontamination vary from technique to technique.

Discrete sediment samples from shallow to moderately deep water can be collected efficiently using polycarbonate (or Lexane) tube. Polycarbonate tube samplers are easy to use, portable, and are a direct method for obtaining sediment samples. The tube is forced into the sediment and then withdrawn using a vacuum/suction technique. Additional sample methods may be utilized if the tube sampling method is not effective in collecting sediments or soils.

Data to be Collected

At each sediment or soil sampling location, a suite of physical variables should be recorded. These variables are intended to locate each probing point and to quantify factors likely to be associated with deposition and accumulation of soft sediments. Variables may include:

- Penetration depth/water depth/recovery depth
- Qualitative rating surface sediment type—silt, sand, gravel, cobble.
- Qualitative assessment of presence or absence of paper residual.
- Sample location river segment type—straight, inside bend, outside bend, depositional zones, floodplain, terrace, etc.
- Presence or absence of benthic organisms (i.e. observation of larval forms present or encrusted on river bottom cobble and rock).
- Noticeable odors, sheens, et cetera.

The following procedures document sampling using a tube, scoop, hand auger, and ponar dredge. Additional soil and sediment SOPs are included in Appendix A.

Tube Sampling Procedure

The following is a procedure to collect sediment or soil samples using polycarbonate tubes.

1. Locate the proposed sample location with a GPS unit.

2. Once at a location, record the water depth (if applicable) from the top of surface water to the top of sediment.

3. Lower the polycarbonate tube until the tube make contacts with the top of the sediment or soil.

4. Gradually force tube into sediment or soil using a post hole driver (or equivalent) to obtain the desired sample depth or until refusal.

5. For sediment samples, fill the remainder of the tube with water and cap to create a vacuum between the top of the sediment and the tubing. Tape cap to tube using either electrical or duct tape to ensure an airtight seal.

6. Pull the tube from the sediment or soil using proper lifting techniques. If the tube becomes stuck, two people may be needed to pull the tube. If the desired recovery is not achieved, remove the sediment or soil within the tube. Move the location over a foot and repeat procedure.

7. When desired sediment or soil retrieval is achieved, place cap on bottom of tubing and secure with tape.

8. With tube held vertically, remove the top cap and cut a hole in the tube with a hack saw just above the top of the sediment to drain off water.

9. Cut off the tube just above the sediment or soil surface and recap the upper end.

10. Label the top cap with the sample location ID, date, time, and "top" description using a permanent marker. Record the date, time, location of the sample, recovery depth, and other salient observations in the field book. Tubes must be kept vertical at an angel greater than 45 degrees.

11. Transport cores to site work area for processing. Cores may need to be on ice if not processed in a reasonable amount of time.

12. Place the polycarbonate tube on a decontaminated worktable and secure. Aluminum foil or plastic sheeting can be used to cover the worktable. For large recoveries, cut the core liner (filled with sediment or soil) lengthwise along opposite sides. (Note: cut through the liner wall without cutting significantly into the core itself. Disturbed sediment or soil adjacent to the liner wall should not be sampled. Also, it is important not to contaminate the undisturbed interior of the core with plastic chips or other debris from the cutting process. The best hand tool available for cutting hard plastic liners is a jig saw being careful to eliminate any of the sample with which the blade has come into contact. For shorter recoveries i.e. less than one foot,

either push the sample out or allow the sample to fall out of the tube while holding vertically.

13. Extend a tape measure along beside the sediment, starting at the original top end of the core.

14. Photograph the core with a digital camera. Photograph the core section in overlapping frames using a small label with core field ID number so that it appears in each frame. Advance the tape measure appropriately for any additional sections of the same core.

15. While the core section is still intact, record a description of the core structure, noting zones of different color, classification, layering, sorting, and sediment or soil type (silt, sand, clay, gravel, etc.).

16. Sample intervals will be determined in the field by CDM or MDEQ PM.

17. Using disposable scoops or by hand with medical gloves, place sediment or soil on aluminum foil or stainless steel bowl and homogenize. (Note that if more than one sample was collected for a location, the sediment or soil from all samples will be combined and homogenized.)

18. Label each container with a unique sample identification number as outlined in Section 3.5.2. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

19. Store the sample bottles on ice or in a refrigerator until transfer shipment to the analytical laboratories.

20. Complete all COC forms and field sheets and record in the field logbook.

21. Decontaminate sampling equipment after use and between sample locations using Alconox and deionized water. Sample equipment may include hacksaw or jigsaw blades. All other equipment i.e. polycarbonate tubing, medical gloves, et cetera are disposable. Sample material not sent to the lab for analysis will be disposed in a municipal landfill.

22. Repeat procedure for each sample location.

Stainless Steel Scoop Sampling

The following is a procedure to collect sediment or soil samples using a stainless steel scoop or shovel.

1. Locate the proposed sample location with a GPS unit.

2. Once at a location, record the water depth (if applicable) from the top of surface water to the top of sediment.

3. Lower the scoop into the sediment or soil being careful not to disturb the surrounding features. Slowly bring the scoop and sample to the surface, again being careful not the let the fine sediment be washed downstream (if sampling sediment).

4. Using disposable scoops or by hand with medical gloves, place sediment or soil on aluminum foil.

5. Extend a tape measure along beside the sediment, starting at the original top end of the core.

6. Photograph the core with a digital camera. Photograph the core section in overlapping frames using a small label with core field ID number so that it appears in each frame. Advance the tape measure appropriately for any additional sections of the same core.

7. While the core section is still intact, record a description of the core structure, noting zones of different color, classification, layering, sorting, and sediment or soil type (silt, sand, clay, gravel, etc.).

8. Sample intervals will be determined in the field by CDM or MDEQ PM.

9. Label each container with a unique sample identification number as outlined in Section 3.5.2. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

10. Store the sample bottles on ice or in a refrigerator until transfer shipment to the analytical laboratories.

11. Complete all COC documents and field sheets and record in the field logbook.

12. Decontaminate sampling equipment after use and between sample locations using Alconox and deionized water. Sample equipment may include stainless steel scoop, sieves, bowl et cetera. All other equipment i.e. polycarbonate tubing, medical gloves, et cetera are disposable. Sample material not sent to the lab for analysis will be disposed of at the sample location.

13. Repeat procedure for each sample location.

Ponar Dredge Sampling

The following is a procedure to collect sediment samples using a ponar dredge.

1. Locate the proposed sample location with a GPS unit.

2. Once at a location, record the water depth (if applicable) from the top of surface water to the top of sediment.

3. Insert the tension spring and slowly the dredge until it reaches the sediment bed. Slowly raise the dredge about two feet and let it drop into the sediment. The force of the dredge in contact with the sediment will release the tension spring and close the dredge on the top 4 to 6 inches of sediment. Pull the dredge out of the water.

4. Open the dredge and place the sediment on aluminum foil.

5. Extend a tape measure along beside the sediment, starting at the original top end of the core.

6. Photograph the core with a digital camera. Photograph the core section in overlapping frames using a small label with core field ID number so that it appears in each frame. Advance the tape measure appropriately for any additional sections of the same core.

7. While the core section is still intact, record a description of the core structure, noting zones of different color, classification, layering, sorting, and sediment or soil type (silt, sand, clay, gravel, etc.).

8. Sample intervals will be determined in the field by CDM or MDEQ PM.

9. Label each container with a unique sample identification number as outlined in Section 3.5.2. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

10. Store the sample bottles on ice or in a refrigerator until transfer shipment to the analytical laboratories.

11. Complete all COC documents and field sheets and record in the field logbook.

12. Decontaminate sampling equipment after use and between sample locations using Alconox and deionized water. Sample equipment may include stainless steel scoop, sieves, bowl et cetera. All other equipment i.e. polycarbonate tubing, medical gloves, et cetera are disposable. Sample material not sent to the lab for analysis will be disposed of at the sample

location.

13. Repeat procedure for each sample location.

Hand Auger Sampling

The following is a procedure to collect sediment or soil samples using a stainless steel hand auger.

1. Locate the proposed sample location with a GPS unit.

2. Once at a location, record the water depth (if applicable) from the top of surface water to the top of soil.

3. Auger though soil at intervals equal to one bucket length. Using a stainless steel spoon or rod, knock out soil from auger onto a piece of aluminum foil, being careful to keep soil in order from top to bottom.

4. Repeat procedure until desired depth achieved or refusal.

5. Extend a tape measure along beside the sediment, starting at the original top end of the core.

6. Photograph the core with a digital camera. Photograph the core section in overlapping frames using a small label with core field ID number so that it appears in each frame. Advance the tape measure appropriately for any additional sections of the same core.

7. While the core section is still intact, record a description of the core structure, noting zones of different color, classification, layering, sorting, and sediment or soil type (silt, sand, clay, gravel, etc.).

8. Sample intervals will be determined in the field by CDM or MDEQ PM.

9. Label each container with a unique sample identification number as outlined in Section 3.5.2. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

10. Store the sample bottles on ice or in a refrigerator until transfer shipment to the analytical laboratories.

11. Complete all COC documents and field sheets and record in the field logbook.

12. Decontaminate sampling equipment after use and between sample locations using Alconox and deionized water. Sample equipment may include stainless steel scoop, sieves, bowl et cetera. All other equipment i.e. polycarbonate

tubing, medical gloves, et cetera are disposable. Sample material not sent to the lab for analysis will be disposed of at the sample location.

13. Repeat procedure for each sample location.

Sample Containers, Preservation Techniques, Quality Control

Refer to Table 3-1 for the specific analyses and sample containers. There is no need for sample preservation, other than, the samples must be stored and shipped on ice. Refer to Table 1-1 for field parameters and QC samples to be collected.

Documentation

Bound field logbooks should be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations shall be documented in the field logbooks. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

5.2 Surface Water Sampling

See Attachment A for collecting depth-integrated surface water samples. This SOP is the same as found in Appendix A of the 2001 LTM Sampling Plan.

5.3 Groundwater Sampling

See Attachment B for SOP on groundwater sampling via low-flow method.

5.4 Water Quality Measurements

See Attachment C for SOP on maintance and operation of water quality meters.

5.5 Sample Custody, Sample Shipping and Data Documentation

See Attachments D and E for SOPs regarding sample custody, packaging and shipping, field book content, and photo documentation.

Section 6
Decontamination Procedures

Procedures to be followed to decontaminate equipment and personnel are described below and in the CDM HSP.

6.1 Personnel Decontamination

Personnel decontamination stations have been set up by the PRPs at designated locations around the site. Personnel will become thoroughly familiar with the decontamination procedure before work begins in exclusion zones. The decontamination procedure is as follows:

- Place equipment and/or samples in segregated equipment drop-areas.
- Remove disposable outer booties (when used).
- Remove hard hat, goggles/safety glasses/face shield.
- Remove disposable nitrile outer gloves.
- Wash hands and face with water and hand soap.

6.2 Equipment Decontamination

Required Materials and Supplies

- 1 – one gallon jug of distilled
- 1 – box or bottle of non-phosphate soap (such as Alconox)
- 1 – 250 foot roll of aluminum foil
- 2 plastic "bottle brushes"
- 5 rolls of paper towels
- 3 – 5-gallon plastic buckets with handles (for washing equipment)
- 1 – 5-gallon bucket lid
- 3 – 30 gallon (approximate) sized garbage bags (for trash)
- 1 pair of disposable nitrile gloves per person

Cleaning Procedure

1) Don nitrile gloves. Using paper towel, wipe off all visible materials that are clinging to the item being decontaminated. The used paper towels should be immediately discarded in a trash bag.

2) In the first 5-gallon bucket, dissolve 1oz. of non-phosphate soap or Alconox to 2 gallons of potable water. If there is no potable water source near the decontamination area, use 2 oz. of soap to 2 gallons of river water.

3) Using the plastic bottlebrush, vigorously scrub the item in the soapy water bucket. After scrubbing, allow the piece to drip over the soapy water bucket for approximately 10 seconds so most of the water used has dripped back into the bucket.

4) Over the second decontamination bucket, rinse the piece once with distilled water. After rinsing, allow the piece to drip over the second decontamination bucket for approximately 10 seconds so most of the water used has dripped back the bucket.

5) Over the third and final bucket, rinse the piece three times with distilled water. After each rinse, allow the piece to drip dry before re-rinsing.

6) After the third and final rinse, allow the piece to air dry on a piece of aluminum foil. After it is air dried, it is now ready to re-use for site work.

7) If the piece is no longer needed, wrap the piece in the aluminum foil it was air dried on for storage.

8) For longer-term storage, small equipment (spatulas, knives, spoons, bowls, etc.) should be stored in resealable bags after wrapping in foil. Larger equipment (Ponar grabs, water samplers, etc.) can be wrapped in larger plastic bags like garbage bags, or stored closed in their field carrying cases.

9) After all equipment has been decontaminated, combine the decon water into a water bucket and properly dispose. Discard the paper towels in the plastic garbage bag immediately.

All reusable non-dedicated equipment (scoops, buckets, core samplers, dredges, bottle sampler) will be decontaminated between samples, and before removal from the site.

6.3 Storage and Disposal of Investigation-Generated Wastes

The sampling activities are expected to generate solid and liquid "waste". Investigative-Derived Waste (IDW) such as gloves, overboots, disposable sampling equipment, etc. may also be generated during sampling activities. The activities, the anticipated type and the planned handling of the wastes are summarized below.

- Soil and sediment: SOLID: Return excess soil or sediment cuttings to the borehole or sampling location after completion of sampling; LIQUIDS: Dispose of decontamination water in 55-gallon DOT approved drums for subsequent characterization and disposal; IDW: Dispose of as common refuse.

- Surface Water and Groundwater sampling: SOLID: None; LIQUIDS: Contain development/purge water in 55-gallon DOT approved drums for subsequent characterization and proper disposal. IDW: Dispose of as common refuse.

The 55-gallon drums generated during purging, sampling, and decontamination will be stored in a secure area on site. Contents will be disposed of based on the results of the characterization. Non-hazardous liquids will be disposed at the site. Any liquids determined to be hazardous will be disposed of properly.

Section 7
Field Quality Control Procedures

To ensure a high level of data quality for sampling events, the following Quality Control (QC) procedures will be performed.

7.1 Soil, Sediment, Surface Water and Groundwater QC Samples

7.1.1 Field Duplicates

Duplicate samples will be collected as follows:

- One duplicate soil or sediment sample for every 20 samples (or portion thereof) collected in the field.
- One duplicate surface water or groundwater sample for every 20 samples (or portion thereof) collected in the field.

7.1.2 Field Blanks

One field blank water sample will be prepared for every 20 surface water or groundwater samples (or portion thereof) collected. Field blanks will be prepared by filling water sample bottles with reagent-grade distilled water from the sampling device (if possible), at the same volume as the sample. These samples will be prepared in close proximity to an actual sample location. This sample location will be recorded in the field log book.

7.1.3 Rinsate Blanks

One rinsate blank for soil or sediment sampling equipment will be collected at the end of each day of sampling if using non-disposable equipment, such as a hand auger. Rinsate blanks will be prepared by pouring laboratory grade distilled/deionized water over the decontaminated non-dedicated soil sampling equipment, and then collecting the water in the sample containers. The FM or STL will record the sample location that preceded collection of the rinsate blank in the field log book.

7.1.4 Matrix Spike and Matrix Spike Duplicates

MS/MSD samples will be collected at a frequency of one per 20 for sediment and soil samples. MS/MSD samples will also be collected at a frequency of one per 20 for surface water and groundwater samples. Matrix spike samples will be denoted by the sample number followed by an '-MS' or '-MSD' suffix on sample labels and COC forms.

Section 8
References

ARCADIS BBL. 2007. Multi-Area Quality Assurance Project Plan (Rev. 00). Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site.

CDM. 2001a. Long Term Monitoring Plan Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site - Sampling Plan.

Attachment A

SOP Water Sampling

Draft

Attachment A
SOP Water Sampling

Collection of Depth-Integrated Surface Water Samples for Organic Contaminant Analysis

A.1 Scope and Application

This standard operating procedure (SOP) describes a method for collecting a 1-liter depth-integrated surface water sample, for organic contaminant analysis. The procedure is applicable for all surface waters (streams, lakes impoundments, wetlands, etc.) within the study area.

The U.S. EPA - Large Lakes Research Station (Grosse Ile, MI), originally developed this procedure for use on the EPA-sponsored Upper Great Lakes Connecting Channel Study (UGLCCS; U.S. EPA, 1988).

A.2 Method Summary

A clean 1-liter amber glass sample bottle is secured within a weighted bottle harness, which has been designed specifically to hold a 1-liter glass sample bottle. The harness is then submerged into the sample water body to be sampled. The harness is then raised and lowered repeatedly to near (within a few inches of) the bottom of the sample water body until the bottle is full.

A.3 Procedure

A.3.1 Materials and Supplies

The following is a list of the minimum quantity of supplies for a surface water-sampling event.

- 6 cases (72 total) 1L Amber glass bottles, with Teflon lined lids
- 51 - 500mL plastic bottles with lids (used for total suspended solids analysis)
- 102 labels
- 2 waterproof pens/markers (MonAmi Acculiner or similar)
- 2 rolls, 3" wide transparent packing tape (for securing labels to bottles)
- 1 bottle harness and 100 feet of ¼ " nylon rope
- 2 metal sheathed thermometers (must be less than 2" in diameter)
- 2 twelve inch long bungee cords (for attaching the plastic jars, see description below)

- 9 ice chests, at least 32 quart capacity each
- Eighteen 7 pound (approximately) bags of ice (2 bags per cooler)
- One gallon jug of distilled water (for blanks and rinsing)
- 2 project specific field books
- 2 field forms
- 1 global positioning system (GPS) unit; one each topographical, road, and project specific map (establishing station position)
- One per team member: waterproof boots/waders/hipboots (for launching boats or hand grabbing water samples without bottle harness)
- Boat/canoe: with 2 oars/paddles and one personal floatation device per team member
- One 100′ roll of 24″ wide bubble wrap (for glass bottles and general packing. Should be the large bubble size. Available at Mailboxes Etc. on Main Street, east of US 131)
- 200 one gallon sized re-sealable (Ziploc type) plastic bags (for all bottles and ice)
- 9 FedEx labels (one for every cooler)
- 9 Chain of Custody forms (one for every cooler)
- 18 Chain of Custody seals (two for every cooler)
- 2 rolls of 3″ Duct tape
- 2 pair of waterproof gloves per team member
- 1 box (100 count minimum) of latex disposable gloves
- Orange safety road vests (one for each team member)
- 2 road barricades (from storage shed)
- Drinking water/snacks/sack lunch: if desired

Bottle harness description: The harness is a metal plate with a flat weight attached to the bottom that is heavy enough to submerge the whole apparatus when empty 1-liter sample bottles (for PCBs) and 500-mL plastic bottles (for TSS) are installed. From the metal base plate, there are three rods equidistant from each other are attached

through the plate. The tops of the rods have closed loops. The nylon lowering rope is threaded through each of the loopholes at the top of the rods and is tied to the last rod loop. When the lowering rope is pulled gently from the top, the rods should collapse around the glass bottle, holding it securely in place. If the bottle is not secure, re-tie the lowering rope.

Attached to one of the three rods is a small clear plastic pipe approximately 3 inches in length and 2 inches in diameter. This is the thermometer reservoir. A thermometer (enclosed in a metal protective sheath) is set in the reservoir. The bulb is submersed in the ambient water that fills the reservoir.

One end of a bungee cord is attached to another rod. The cord is then wrapped twice a round the 1 liter glass and 500-ml plastic bottles and the end secured to the harness.

A.3.2 Water Sampling Procedure

[Note: steps marked with "**" denote general descriptions of activities that will vary from project to project, the details of which are described in detail in the project-specific Field Sampling Plan.]

- Establish sampling position on station and record location in the field notebook.** Appropriate procedures will include use of a global positioning system, triangulation on local landmarks, careful notations on a detailed map, etc.

- Fill out label with sample number (see Appendix H for correct nomenclature), station, date, time etc. with waterproof pen/marker and affix label to both amber and plastic bottles. Wrap labels with clear waterproof tape all the way around the bottles.

- Secure amber bottle in middle of bottle harness. Secure plastic bottle near thermometer reservoir with attached bungee cord. Uncap both bottles.

- Lower the bottle into the water column and begin raising and lowering the bottle and harness up and down through the water column until both bottles are full. This will typically take between 2 and 4 minutes. Do not to let the bottle come into contact with the sediment, or sample the surface water film.

- When both sample bottles are full, pull the harness from the water, recap both bottles and remove them from the harness. Record water temperature in field book after capping bottles.

A.3.3 Packaging Procedure

- Place each amber bottle into a resealable bag. One 1-liter bottle per bag. Put the plastic bottles in a bag also. 3 – 500 ml plastic bottles per bag. Wrap each bagged amber bottle once in bubble wrap, ensuring adequate coverage to the bottle neck area. Use the transparent tape to hold bubble wrap in place.

- Fill several one-gallon resealable bags with ice. Each bag of ice then needs to be placed within a second empty resealable bag (Double bagging).

- Place bottles standing up in the ice chest. Distribute double-bagged ice packs around bottles to ensure that all bottles stay cool. When cooler is full, fill empty spaces with extra bubble wrap or other packing material (Styrofoam peanuts if available).

- Ensure each cooler has its own COC form filled out for its contents only and put in a resealable waterproof bag. Have a coworker double-check the COC before it is put in the bag. Remember to keep the pink copy of the COC. Tape the bag to the inside lid of the ice chest.

- If the ice chest will not arrive the next day at the desired location, hold the coolers at the storage shed and ice daily.

- Close the ice chest and affix COC seals on opposite sides of the cooler. Seal cooler with the packing or duct tape. At a minimum, wrap tape two times around each end and around the lip of the ice chest. Also make sure that if the ice chest has a drainage plug that it is sealed as well.

- Complete the FedEx form ensuring that all the correct boxes are marked, including those for weekend delivery if required. The address for weekday delivery is Bob Wagner (Phone: 518.346.4592), NEA Labs, 2190 Technology Drive in Schenectady, New York, 12308. Use 1785-28963-T2A1.RPT as "Your Internal Billing Reference". The samples should be sent FedEx Priority Overnight and billed to the Sender. Weekend delivery should be avoided. If weekend delivery is required, it should be cleared through the Project Manager. Also, confirm the availability of weekend delivery to the recipient as well as their correct weekend address prior to shipment. Have a coworker double check the paperwork and keep the top copy.

- After arriving at FedEx service center (such as MailBoxes Etc. at 5047 West Main Street in Kalamazoo), affix a clear envelope to the lid of the ice chest. Place FedEx form inside. Do not remove narrow strip on top of envelope. If Saturday or Sunday delivery is desired, attach appropriate stickers. Also attach "Fragile" stickers to each ice chest.

- Warning: The last pickup for the day at most FedEx service centers is 5 P.M. If this can not be met, packages can be dropped off at the FedEx terminal in Portage until 7 P.M. It is located at 6180 Corporate Avenue off of Sprinkle Road.

- All field notes, chain of custody forms and FedEx slips should be stapled to each other and given to the scheduler in the Detroit office.

A.4 Field QA/QC Procedures

- To minimize sample contamination, take care not to allow the sample bottle and harness to come into contact with the bottom sediment, or to sample the surface film.

- Details of the field QA/QC procedures will be project-specific and are described in the project Field Sampling Plan. Typical field QA/QC operations include:

  - Collection of duplicate water samples and matrix spike/matrix spike duplicates, at regular intervals (a certain number per day or per water body sampled, or 1 per 10 or 20 samples).

  - Equipment rinsate blanks, of the filler caps.

A.5 Health and Safety

Normal water safety precautions must always be observed, including wearing life vests, taking care to avoid hypothermia or heatstroke, etc.

A.6 References Cited

U.S. EPA. 1988. *Upper Great Lakes Connecting Channels Study – Volume II – Final Report*. U.S. EPA Great Lakes National Program Office, Chicago, IL. 626 pp.

Attachment B

SOP Low-Flow Water Sampling

Draft

Attachment B
SOP Low Flow Water Sampling

B.1 Objective

The purpose of this standard operating procedure (SOP) is to define requirements for the collection of groundwater samples using a low-flow, minimal drawdown method.

B.2 Background

Collection of groundwater samples from monitoring wells on or near a hazardous waste site may be required to characterize the nature and extent of contamination. The objective of low-flow sampling is to minimize the drawdown of an aquifer and the disturbance within a well screen so that the groundwater sample being collected is most representative of the water passing through the well screen.

Methods used for the collection of groundwater samples using low-flow techniques may include a variety of pumps, such as a peristaltic, bladder, submersible, and gas-driven pumps, depending on site conditions. The bailer method is inappropriate for low flow sampling.

B.3 General Responsibilities

Site Manager - The site manager is responsible for ensuring that field personnel are trained in the use of this procedure and for verifying that groundwater samples are collected in accordance with this procedure.

Field Team Leader - The field team leader is responsible for ensuring that sampling efforts are conducted in accordance with this procedure and any associated SOPs.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

B.4 Required Equipment

- Site-specific plans
- Field logbook
- Indelible black ink pens and markers
- Labels and appropriate forms/documentation for sample shipment
- Sample chain-of-custody forms
- Insulated cooler and waterproof sealing tape (strapping tape)
- Plastic zip-top bags

- Ice double-bagged in plastic zip-top bags
- Multi-parameter water quality meter (YSI 556 or equivalent)
- Hach 2100p (or equivalent)
- Calibration solutions for meters
- Water level meter
- Graduated cup
- Two 5-gallon bucket
- 200' tape measure
- Flow through cell for measurement of water quality parameters
- Plastic sheeting (4-mil thickness)
- Appropriate size disposable tubing
- Peristaltic, bladder, submersible, and gas-driven pump
- Tubing cutter
- Latex or appropriate gloves
- Filtering apparatus, if required
- Appropriate sample containers with labels and preservatives, as required
- Photoionization detector (PID) or equivalent and other instruments as required by the site-specific health and safety plan
- Decontamination supplies as required
- Lab grade deionized water for equipment blank samples
- Personal protective clothing and equipment as required by the site-specific health and safety plan

B.5 Procedures

1. Review site-specific health and safety plan and project plans before initiating sampling activity.

2. Calibrate multi-parameter water quality meter and turbidity meter according to manufacters recommendations and site QAPP. Check water level meter by dipping the probe in tap water and checking to ensure that the signaling device responds to probe submergence. Record this information in the field logbook or on the appropriate form.

3. Don personal protective clothing and equipment as specified in the site-specific health and safety plan. All field equipment will be calibrated, tested, or checked for proper functioning before use per the manufacturer's instructions.

4. Prepare the site for sample acquisition. If required, cover the ground surface around the wellhead with plastic sheeting. Arrange the required decontaminated sampling and monitoring equipment for convenient use. If onsite decontamination is required, arrange the necessary supplies in a nearby but separate location, away from the wellhead (i.e., in an exclusion zone).

5. Open the well and note the condition of the casing and cap. Immediately check for organic vapors using a PID or flame ionization detector as appropriate. Refer to the site health and safety plan for the required monitoring and frequencies.

6. Determine the static water level using an electrical water level indicator after adequate time is allowed for the well to vent. The probe shall then be lowered slowly into the well until contact with the water surface is indicated. The electrical tape reading is made at the measuring point. Take a second and third check reading to verify the measurement before completely withdrawing the tape from the well. Record this information in the field logbook or on the appropriate form.

7. Independent electrical tape measurements of static water levels using the tape shall agree within 0.01 foot for depths of less than about 200 feet. Decontaminate the electrical tape according to QAPP before proceeding to the next well to minimize cross contamination. It may be necessary to check the electrical tape length with a graduated steel tape after the line has been used for a long period of time (at least annually) or after it has been pulled hard in attempting to free the line.

8. Sample pump or tubing may either be dedicated or disposable. If a dedicated pump or tubing is to be used (preferred method), pull dedicated intake up to the correct sample depth. A mark should be made on the tubing so any sampler can be able to determine the correct sample depth without pulling the entire length of tubing and/or pump from the well. Placement of the pump or tubing intake should be in the middle or at the

top of the well screen to minimize the entrainment of solids that may have collected in the bottom of the well.

9. If sample pump and/or tubing is disposable, the tubing and/or pump should be installed with minimal disturbance to the water column. In some cases, tubing and/or pump should be installed several days before sampling so that any solids disturbed during installation will have adequate time to settle before sampling. The correct intake depth needs to be determined by subtracting one-half the screen length from the total well depth. Measure out the correct tubing that is needed, plus an additional few feet so the tubing can extend over the casing. Very slowly lower the tubing and/or pump into the well to the correct intake depth. Mark tubing location with permanent marker.

10. Connect well tubing to pump. If using a peristaltic pump, connect the well tubing to the flexible hose tubing used in the pump. Cut a second piece of well tubing a few feet in length and connect bottom port to the flow-through cell of the multi-parameter water quality meter. Connect a third piece of tubing to the top port of the flow-through cell and let the water drain into a 5-gallon bucket.

11. Turn pump on. Record start time in field logbook or form. Measure flow rate as soon as possible using graduated cup and stopwatch. If flow faster or slower than desired rate, decrease or increase the pumping rate accordingly. Low flow purging and sampling should be conducted at flows less than 500 mL/min, usually between 100 and 500 mL/min. Continually check on flow rate throughout the purging and sampling process. The goal of low-flow, minimal drawdown sampling is to have less than 0.1 meter of drawdown during purging.

12. Record the water level, flow rate, and meter readings (pH, water temperature, conductivity, oxidation reduction potential, dissolved oxygen) in the field logbook or form every three to five minutes until parameters stabilize. Wipe Hach vial with Kim-wipe to clean vial before measure turbidity. Stabilization occurs when three consecutive readings are within the ranges below (note that dissolved oxygen and turbidity are usually the last to stabilze).

    - Turbidity ± 10% and < 5 NTUs
    - pH is ± 0.1
    - DO ± 10%
    - conductivity ± 3%

- ORP ± 10 mV

13. After all parameters are stable, remove tubing from the bottom port of the flow through cell and begin to fill sample bottles. Bottles are usually filled in the order below. Since PCBs are the contaminant of concern at the site, the PCB bottle will be filled up first immediately after parameter stabilization to ensure that parameters do not drift over time, especially turbidity. Refer to QAPP for specifics on container type and preservative for each chemical group.

    - PCBs
    - Volatile organic compounds (VOCs)
    - Purgeable organic carbon (POC)
    - Purgeable organic halogens (POX)
    - Total organic halogens (TOX)
    - Total organic carbon (TOC)
    - Semivolatile organic compounds (SVOCs) and pesticides
    - Dissolved metals
    - Cyanide
    - Sulfate and chloride
    - Nitrate and ammonia
    - Radionuclides

14. When filling duplicate samples, fill 1/3 of the original sample bottle, then 1/3 of the duplicate sample bottle. Continue filling each bottle in thirds to ensure more accurate duplicate results.

15. After a sample container is filled, wipe the outer surfaces of the sample containers clean with a Kim-wipe or clean paper towel. Additional sample bottle decontamination may be appropriate in some cases.

16. Properly label all containers according to QAPP. Seal label on container using clear packing tape.

17. Place sample containers in individual zip-top plastic bags and seal the bags (if required by site-specific plans).

18. Immediately pack all sample containers that require a 4°C preservation on ice in coolers (refer to the site-specific field sampling plan).

19. Record analytes and volumes collected, time and date of collection, sample identification, and all other pertinent sample information into the field logbook. Prepare chain-of-custody forms according to site-specific plans and QAPP.

20. When all applicable samples have been collected, record static water level and the total well depth. Turn the pump off and record the stop time. If required by the site-specific field sampling plan, obtain required field measurements such as water level, temperature, conductivity, pH, oxidation potential, turbidity, and dissolved oxygen measurements immediately after samples have been collected and record them in the field logbook.

21. Decontaminate sampling equipment as needed, according to site-specific plans and QAPP.

22. Close and lock the well cover. Clean up the area and place disposable materials (plastic sheeting, gloves, Tyvek®) in the designated waste receptacle. Dispose of purged water according to QAPP.

B.6 Restrictions/Limitations

Careful sampling for VOC analysis or for analysis of any other compound(s) that may be degraded by aeration is necessary to minimize sample disturbance and, hence, analyte loss. The representativeness of this sample, however, is difficult to determine because the collected sample represents a single point, is not homogenized, and has been disturbed.

B.7 References

Office of Solid Waste and Emergency Response. 1996. RCRA Ground Water Issue: Low-Flow (Minimal Drawdown) Ground-water Sampling Procedures, EPA/540/S-95/504. April.

Attachment C

SOP 5-1 Water Quality Measurements

Draft

Control of Measurement and Test Equipment

SOP 5-1
Revision: 8
Date: March 2007

Prepared: Dave Johnson

Technical Review: Steve Guthrie

E-Signed by Michael C. Malloy
VERIFY authenticity with ApproveIt

QA Review: Jo Nell Mullins

Approved:

Signature/Date

E-Signed by P. Michael Schwan
VERIFY authenticity with ApproveIt

Issued:

Signature/Date

1.0 Objective

The objective of this standard operating procedure (SOP) is to establish the baseline requirements, procedures, and responsibilities inherent to the control and use of all measurement and test equipment (M&TE). Contractual obligations may require more specific or stringent requirements that must also be implemented.

2.0 Background

2.1 Definitions

Traceability - The ability to trace the history, application, or location of an item and like items or activities by means of recorded identification.

2.2 Associated Procedures

- CDM Federal Technical SOP 4-1, *Field Logbook Content and Control*
- CDM Quality Procedures (QPs) 2.1 and 2.3
- Manufacturer's operating and maintenance and calibration procedures

2.3 Discussion

M&TE may be government furnished (GF), rented or leased from an outside vendor, or purchased. It is essential that measurements and tests resulting from the use of this equipment be of the highest accountability and integrity. To facilitate that, the equipment shall be used in full understanding and compliance with the instructions and specifications included in the manufacturer's operations and maintenance and calibration procedures and in accordance with any other related project-specific requirements.

3.0 Responsibilities

All staff with responsibility for the direct control and/or use of M&TE are responsible for being knowledgeable of and understanding and implementing the requirements contained herein as well as any other related project-specific requirements.

The project manager (PM) or designee (equipment coordinator, quality assurance coordinator, field team leader, etc.) is responsible for initiating and tracking the requirements contained herein.

***Note*:** Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Requirements for M&TE

- Determine and implement M&TE related project-specific requirements
- The maintenance and calibration procedures must be followed when using M&TE
- Obtain the maintenance and calibration procedures if they are missing or incomplete
- Attach or include the maintenance and calibration procedures with the M&TE
- Prepare and record maintenance and calibration in an equipment log or a field log as appropriate (Figure 1)
- Maintain M&TE records
- Label M&TE requiring routine or scheduled calibration (when required)
- Perform maintenance and calibration using the appropriate procedure and calibration standards
- Identify and take action on nonconforming M&TE

5.0 Procedures

5.1 Determine if Other Related Project-Specific Requirements Apply

For all M&TE:
The PM or designee shall determine if M&TE related project-specific requirements apply. If M&TE related project-specific requirements apply, obtain a copy of them and review and implement as appropriate.

5.2 Obtain the Operating and Maintenance and Calibration Documents

For GF M&TE that is to be procured:
Requisitioner - Specify that the maintenance and calibration procedures be included.

For GF M&TE that is acquired as a result of a property transfer:
Receiver - Inspect the M&TE to determine whether maintenance and calibration procedures are included with the item. If missing or incomplete, order the appropriate documentation from the manufacturer.

For M&TE that is to be rented or leased from an outside vendor:
Requisitioner - Specify that the maintenance and calibration procedures, the latest calibration record, and the calibration standards certification be included. If this information is not delivered with the M&TE, ask the procurement division to request it from the vendor.

5.3 Prepare and Record Maintenance and Calibration Records

For all M&TE:
PM or Designee - Record all maintenance and calibration events in a field log unless other project-specific requirements apply.

For GF M&TE only (does not apply to rented or leased M&TE):
If an equipment log is a project specific requirement, perform the following:

Receiver - Notify the PM or designee for the overall property control of the equipment upon receipt of an item of M&TE.

PM or Designee and User:
- Prepare a sequentially page numbered equipment log for the item using the maintenance and calibration form (or equivalent) (Figure 1).
- Record all maintenance and calibration events in an equipment log.

5.4 Label M&TE Requiring Calibration

For GF M&TE only (does not apply to rented or leased M&TE):
If calibration labeling is a project specific requirement, perform the following:

PM or Designee:
- Read the maintenance and calibration procedures to determine the frequency of calibration required.
- If an M&TE item requires calibration before use, affix a label to the item stating “Calibrate Before Use.”
- If an M&TE item requires calibration at other scheduled intervals, e.g., monthly, annually, etc., affix a label listing the date of the last calibration, the date the item is next due for a calibration, the initials of the person who performed the calibration, and a space for the initials of the person who shall perform the next calibration.

5.5 Operating, Maintaining or Calibrating an M&TE Item

For all M&TE:
PM or Designee and User - Operate, maintain, and calibrate M&TE in accordance with the maintenance and calibration procedures. Record maintenance and calibration actions in the equipment log or field log.

5.6 Shipment

For GF M&TE:
Shipper - Inspect the item to ensure that the maintenance and calibration procedures are attached to the shipping case, or included, and that a copy of the most recent equipment log entry page (if required) is included with the shipment. If the maintenance and calibration procedures and/or the current equipment log page (if required) is missing or incomplete, do not ship the item. Immediately contact the PM or designee and request a replacement.

For M&TE that is rented or leased from an outside vendor:
Shipper - Inspect the item to ensure that the maintenance and calibration procedures and latest calibration and standards certification records are included prior to shipment. If any documentation is missing or incomplete, do not ship the item. Immediately contact the procurement division and request that they obtain the documentation from the vendor.

5.7 Records Maintenance

For GF M&TE:
PM or Designee - Create a file upon the initial receipt of an item of M&TE or calibration standard. Organize the files by contract origin and by M&TE item and calibration standard. Store all files in a cabinet, file drawer, or other appropriate storage media at the pertinent warehouse or office location.

Receiver - Forward the original packing slip to the procurement division and a photocopy to the PM or designee.

PM or Designee and User:
- Maintain all original documents in the equipment file except for the packing slip and field log.
- File the photocopy of the packing slip in the M&TE file.
- Record all maintenance and calibration in an equipment log or field log (as appropriate). File the completed equipment logs in the M&TE records. Forward completed field logs to the PM for inclusion in the project files.

For M&TE rented or leased from an outside vendor:
Receiver - Forward the packing slip to the procurement division.

User:
- Forward the completed field log to the PM for inclusion in the project files.
- Retain the most current maintenance and calibration record and calibration standards certifications with the M&TE item and forward previous versions to the PM for inclusion in the project files.

5.8 Traceability of Calibration Standards

For all items of M&TE:
PM or Designee and User:
- When ordering calibration standards, request nationally recognized standards as specified or required. Request commercially available standards when not otherwise specified or required. Or, request standards in accordance with other related project-specific requirements.
- Require certifications for standards that clearly state the traceability.
- Require Material Safety Data Sheets to be provided with standards.
- Note standards that are perishable and consume or dispose of them on or before the expiration date.

5.9 M&TE That Fails Calibration

For any M&TE item that cannot be calibrated or adjusted to perform accurately:
PM or Designee
- Immediately discontinue use and segregate the item from other equipment. Notify the appropriate PM and take appropriate action in accordance with the CDM QP 2.3 for nonconforming items.
- Review the current and previous maintenance and calibration records to determine if the validity of current or previous measurement and test results could have been affected and notify the appropriate PM(s) of the results of the review.

6.0 Restrictions/Limitations

On an item-by-item basis, exemptions from the requirements of this SOP may be granted by the Headquarters health and safety manager and/or Headquarters quality assurance director. All exemptions shall be documented by the grantor and included in the equipment records as appropriate.

7.0 References

CDM Federal Programs Corporation. 2007. *Quality Assurance Manual.* Rev. 11.

CDM Federal Programs Corporation. 2005. *Government Property Manual*. Rev. 3.

Figure 1

CDM
A subsidiary of Camp Dresser & McKee Inc.

Maintenance and Calibration

Date: Time: (a.m./p.m.)

Employee Name: Equipment Description:

Contract/Project: Equipment ID No.:

Activity: Equipment Serial No.:

Maintenance

Maintenance Performed:

Comments:

Signature: Date:

Calibration/Field Check

Calibration Standard: Concentration of Standard:

Lot No. of Calibration Standard: Expiration Date of Calibration Standard:

Pre-Calibration Reading: Post-Calibration Reading:

Additional Readings: Additional Readings:

Additional Readings: Additional Readings:

Pre-Field Check Reading: Post-Field Check Reading:

Adjustment(s):

Calibration: □ Passed □ Failed

Comments:

Signature: Date:

YSI Environmental

YSI 556 Multiparameter System

Versatile, multiparameter handheld instrument

Rugged and reliable, the YSI 556 MPS (Multiprobe System) combines the versatility of an easy-to-use, easy-to-read handheld unit with all the functionality of a multiparameter system.

- Simultaneously measures dissolved oxygen, pH, conductivity, temperature, and ORP
- Field-replaceable electrodes
- Compatible with EcoWatch® for Windows® data analysis software
- Stores over 49,000 data sets, time and date stamped, interval or manual logging
- Three-year warranty on the instrument; one-year on the probes
- GLP assisting, records calibration data in memory
- Available with 4, 10, and 20-m cable lengths
- IP-67, impact-resistant, waterproof case
- Easy-to-use, screw-on cap DO membranes
- RS-232 interface for PC connection

The 556 has multiple language capabilities and graphing!

Options to Fit Your Applications!

- Battery Options – The unit is powered by alkaline batteries or an optional rechargeable battery pack with quick-charge feature.
- Optional Barometer – Internal barometer can be user-calibrated and displayed along with other data, used in dissolved oxygen calibrations, and logged to memory for tracking changes in barometric pressure. (Choose 556-02)
- Optional Flow Cell - The 5083 flow cell can be used for ground water applications or anytime water is pumped for sampling.
- Carrying Case – The instrument comes standard with YSI 5061, a soft-sided carrying case with enough space for the 556, a 20-meter cable, and calibrating supplies. An optional 5080 hard-sided carrying case is also available.
- Confidence Solution® - Quality assurance ensured. Quickly check conductivity, pH, and ORP readings with one solution.

Pure Data for a Healthy Planet.®

A rugged, cost-effective multiparameter handheld system designed for the field!

5563 MPS Sensor Specifications

Dissolved Oxygen (% saturation)	Sensor Type	Steady state polarographic
	Range	0 to 500% air saturation
	Accuracy	0 to 200% air saturation, ± 2% of the reading or ±2% air saturation, whichever is greater; 200 to 500% air saturation, ± 6% of the reading
	Resolution	0.1% air saturation
Dissolved Oxygen (mg/L)	Sensor Type	Steady state polarographic
	Range	0 to 50 mg/L
	Accuracy	0 to 20 mg/L, ± 2% of the reading or ±0.2 mg/L, whichever is greater; 20 to 50 mg/L, ± 6% of the reading
	Resolution	0.01 mg/L
Temperature	Sensor Type	YSI Temperature Precision™ thermistor
	Range	-5 to 45°C
	Accuracy	± 0.15°C
	Resolution	0.1°C
Conductivity	Sensor Type	4-electrode cell with autoranging
	Range	0 to 200 mS/cm
	Accuracy	± 0.5% of reading or ± 0.001 mS/cm; whichever is greater (4-meter cable) ± 1.0% of reading or ± 0.001 mS/cm; whichever is greater (20-meter cable)
	Resolution	0.001 mS/cm to 0.1 mS/cm (range-dependent)
Salinity	Sensor Type	Calculated from conductivity and temperature
	Range	0 to 70 ppt
	Accuracy	± 1.0% of reading or ±0.1 ppt, whichever is greater
	Resolution	0.01 ppt
pH (optional)	Sensor Type	Glass combination electrode
	Range	0 to 14 units
	Accuracy	±0.2 units
	Resolution	0.01 units
ORP (optional)	Sensor Type	Platinum button
	Range	-999 to +999 mV
	Accuracy	± 20 mV
	Resolution	0.1 mV
Total Dissolved Solids (TDS)	Sensor Type	Calculated from conductivity (variable constant, default 0.65)
	Range	0 to 100 g/L
	Resolution	4 digits
Barometer (optional)	Range	500 to 800 mm Hg
	Accuracy	± 3 mm Hg within ± 15°C temperature range from calibration point
	Resolution	0.1 mm Hg

YSI 556 Instrument Specifications

Size	11.9 cm width x 22.9 cm lenth (4.7 in. x 9 in.)
Weight with batteries	2.1 lbs. (916 grams)
Power	4 alkaline C-cells; optional rechargeable pack
Cables	4-, 10-, and 20-m (13.1, 32.8, 65.6 ft.) lengths
Warranty	3-year instrument; 1-year probes and cables
Communication Port	RS-232 Serial
Data Logger	49,000 data sets, date and time stamp, manual or logging, with user-selectable intervals

556 Ordering Information (Order all items separately)

556-01	Instrument (with 5061 large, soft-sided carrying case)
556-02	Instrument with barometer option (with 5061 carrying case)
5563-4	4-m cable and DO/temp/conductivity
5563-10	10-m cable and DO/temp/conductivity
5563-20	20-m cable and DO/temp/conductivity
5564	pH Probe for any 5563 cable
5565	pH/ORP Probe for any 5563 cable
6118	Rechargeable battery pack kit (includes battery, adapter, charger)
614	Ultra clamp, C-clamp mount
616	Charger, cigarette lighter
4654	Tripod (small tripod for instrument)
5060	Small carrying case, soft-sided (fits instrument and 4-m cable)
5065	Form-fitted carrrier with shoulder strap
5080	Small carrying case, hard-sided (fits instrument, 4-m cable, flow cell, batteries, membrane kit, calibration bottles)
5083	Flow cell
5085	Hands-free harness
5580	Confidence Solution® (insure probe accuracy with a simple field-check for conductivity, pH, and ORP)

The 5080 carrying case with 556, 5563-4 cable, and 5083 flow cell.

To order, or for more information, contact YSI Environmental.

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Attachment D

SOP 2-1 Packaging and Shipping Environmental Samples

Draft

Packaging and Shipping Environmental Samples

SOP 2-1
Revision: 3
Date: March 2007

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VERIFY authenticity with ApproveIt

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Signature/Date

1.0 Objective

The objective of this SOP is to outline the requirements for the packaging and shipment of environmental samples. Additionally, Sections 2.0 through 7.0 outline requirements for the packaging and shipping of regulated environmental samples under the Department of Transportation (DOT) Hazardous Materials Regulations, the International Air Transportation Association (IATA), and International Civil Aviation Organization (ICAO) Dangerous Goods Regulations for shipment by air and applies only to domestic shipments. This SOP does not cover the requirements for packaging and shipment of equipment (including data loggers and self-contained breathing apparatus [SCBAs] or bulk chemicals that are regulated under the DOT, IATA, and ICAO.

1.1 Packaging and Shipping of All Samples

This standard operating procedure (SOP) applies to the packaging and shipping of all environmental samples. If the sample is preserved or radioactive, the following sections may also be applicable.

Section 2.0 - Packaging and Shipping Samples Preserved with Methanol
Section 3.0 - Packaging and Shipping Samples Preserved with Sodium Hydroxide
Section 4.0 - Packaging and Shipping Samples Preserved with Hydrochloric Acid
Section 5.0 - Packaging and Shipping Samples Preserved with Nitric Acid
Section 6.0 - Packaging and Shipping Samples Preserved with Sulfuric Acid
Section 7.0 - Packaging and Shipping Limited-Quantity Radioactive Samples

1.2 Background

1.2.1 Definitions

Environmental Sample - An aliquot of air, water, plant material, sediment, or soil that represents the contaminant levels on a site. Samples of potential contaminant sources, like tanks, lagoons, or non-aqueous phase liquids are normally not "environmental" for this purpose. This procedure applies only to environmental samples that contain less than reportable quantities for any foreseeable hazardous constituents according to DOT regulations promulgated in 49 CFR - Part 172.101 Appendix A.

Custody Seal - A custody seal is a narrow adhesive-backed seal that is applied to individual sample containers and/or the container (i.e., cooler) before offsite shipment. Custody seals are used to demonstrate that sample integrity has not been compromised during transportation from the field to the analytical laboratory.

Inside Container - The container, normally made of glass or plastic, that actually contacts the shipped material. Its purpose is to keep the sample from mixing with the ambient environment.

Outside Container - The container, normally made of metal or plastic, that the transporter contacts. Its purpose is to protect the inside container.

Secondary Containment - The outside container provides secondary containment if the inside container breaks (i.e., plastic overpackaging if liquid sample is collected in glass).

Excepted Quantity - Excepted quantities are limits to the mass or volume of a hazardous material in the inside and outside containers below which DOT, IATA, ICAO regulations do not apply. The excepted quantity limits are very low. Most regulated shipments will be made under limited quantity.

Limited Quantity - Limited quantity is the maximum amount of a hazardous material below which there are specific labeling or packaging exceptions.

Performance Testing - Performance testing is the required testing of outer packaging. These tests include drop and stacking tests.

Qualified Shipper - A qualified shipper is a person who has been adequately trained to perform the functions of shipping hazardous materials.

1.2.2 Associated Procedures

- CDM Federal SOP 1-2, *Sample Custody*

1.2.3 Discussion

Proper packaging and shipping is necessary to ensure the protection of the integrity of environmental samples shipped for analysis. These shipments are potentially subject to regulations published by DOT, IATA, or ICAO. Failure to abide by these rules places both CDM and the individual employee at risk of serious fines. The analytical holding times for the samples must not be exceeded. The samples shall be packed in time to be shipped for overnight delivery. Make arrangements with the laboratory before sending samples for weekend delivery.

1.3 Required Equipment

- Coolers with return address of the appropriate CDM office
- Heavy-duty plastic garbage bags
- Plastic zip-type bags, small and large
- Clear tape
- Nylon reinforced strapping tape
- Duct tape
- Vermiculite (or an equivalent nonflammable material that is inert and absorbent)*
- Bubble wrap (optional)
- Ice
- Custody seals
- Completed chain-of-custody record or contract laboratory program (CLP) custody records, if applicable
- Completed bill of lading
- "This End Up" and directional arrow labels

*Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

1.4 Packaging Environmental Samples

The following steps must be followed when packing sample bottles and jars for shipment:

1. Verify the samples undergoing shipment meet the definition of "environmental sample" and are not a hazardous material as defined by DOT. Professional judgment and/or consultation with qualified persons such as the appropriate health and safety coordinator or the health and safety manager shall be observed.

2. Select a sturdy cooler in good repair. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler. Line the cooler with a large heavy-duty plastic garbage bag.

3. Be sure the caps on all bottles are tight (will not leak); check to see that labels and chain-of-custody records are completed properly (SOP 1-2, Sample Custody).

4. Place all bottles in separate and appropriately sized plastic zip-top bags and close the bags. Up to three VOA vials may be packed in one bag. Binding the vials together with a rubber band on the outside of the bag, or separating them so that they do not contact each other, will reduce the risk of breakage. Bottles may be wrapped in bubble wrap. Optionally, place three to six VOA vials in a quart metal can and then fill the can with vermiculite or equivalent. ***Note***: Trip blanks must be included in coolers containing VOA samples.

5. Place 2 to 4 inches of vermiculite (or equivalent) into a cooler that has been lined with a garbage bag, and then place the bottles and cans in the bag with sufficient space to allow for the addition of packing material between the bottles and cans. It is preferable to place glass sample bottles and jars into the cooler vertically. Glass containers are less likely to break when packed vertically rather than horizontally.

6. While placing sample containers into the cooler, conduct an inventory of the contents of the shipping cooler against the chain-of-custody record. The chain-of-custody with the cooler shall reflect only those samples within the cooler.

7. Put ice in large plastic zip-top bags (double bagging the zip-tops is preferred) and properly seal. Place the ice bags on top of and/or between the samples. Several bags of ice are required (dependant on outdoor temperature, staging time, etc.) to maintain the cooler temperature at approximately 4° Celsius (C) if the analytical method requires cooling. Fill all remaining space between the bottles or cans with packing material. Securely fasten the top of the large garbage bag with fiber or duct tape.

8. Place the completed chain-of-custody record or the CLP traffic report form (if applicable) for the laboratory into a plastic zip-top bag, seal the bag, tape the bag to the inner side of the cooler lid and close the cooler.

9. The cooler lid shall be secured with nylon reinforced strapping tape by wrapping each end of the cooler a minimum of two times. Attach a completed chain-of-custody seal across the opening of the cooler on opposite sides. The custody seals shall be affixed to the cooler with half of the seal on the strapping tape so that the cooler cannot be opened without breaking the seal. Complete two more wraps around with fiber tape and place clear tape over the custody seals.

10. The shipping container lid must be marked **"THIS END UP"** and arrow labels that indicate the proper upward position of the container shall be affixed to the cooler. A label containing the name and address of the shipper (CDM) shall be placed on the outside of the container. Labels used in the shipment of hazardous materials (such as Cargo Only Air Craft, Flammable Solids, etc.) are not permitted on the outside of containers used to transport environmental samples and shall not be used. The name and address of the laboratory shall be placed on the container, or when shipping by common courier, the bill of lading shall be completed and attached to the lid of the shipping container.

2.0 Packaging and Shipping Samples Preserved with Methanol

2.1 Containers

- The maximum volume of methanol in a sample container is limited to 30 ml.
- The sample container must not be full of methanol.

2.2 Responsibility

It is the responsibility of the qualified shipper to:

- Ensure that the samples undergoing shipment contain no other contaminant that meets the definition of "hazardous material" as defined by DOT
- Determine the amount of preservative in each sample so that accurate determination of quantities can be made

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

2.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Inner packing may consist of glass or plastic jars
- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test
- Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
- Class 3 flammable liquid labels
- Orientation labels
- Consignor/consignee labels

2.4 Packaging Samples Preserved with Methanol

The following steps are to be followed when packaging limited-quantity sample shipments:

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape before sampling.
- At a minimum the label must contain:
  - Project name
  - Project number
  - Date and time of sample collection
  - Sample location
  - Sample identification number
  - Collector's initials
  - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)
- Wrap each container (40-ml VOA vials) in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
- Place wrapped containers inside a polyethylene bottle filled with vermiculite; seal the bottle. (Maximum of 4 VOA vials will fit inside a 500-ml wide-mouth polyethylene bottle.)
- Total volume of methanol per shipping container must not exceed 500 ml.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tieing or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

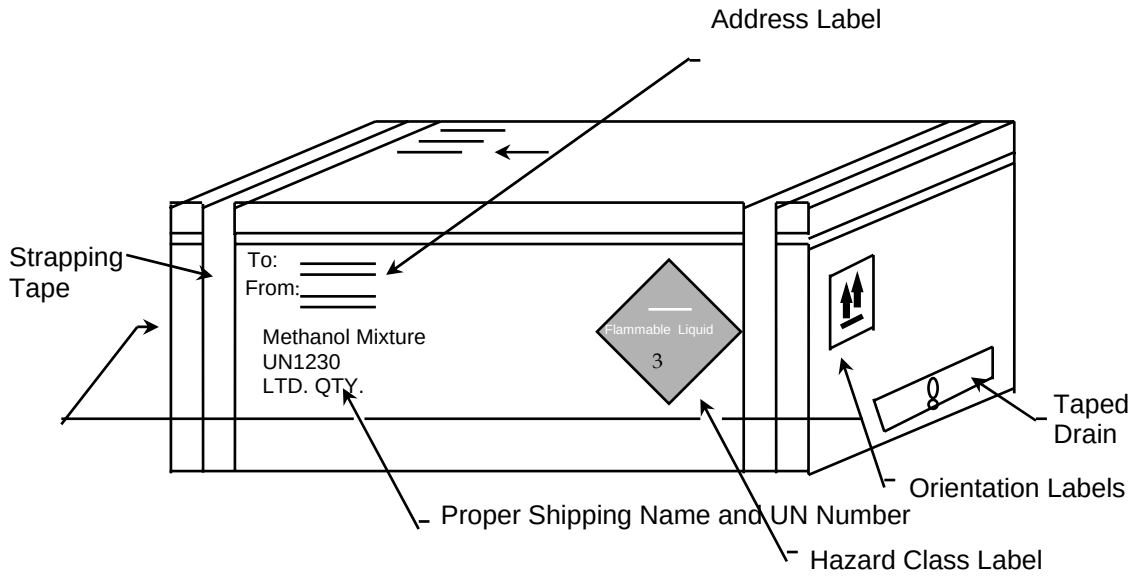
  Methanol Mixture
  UN1230
  LTD. QTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Flammable Liquid label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.

Note: No marking or labeling can be obscured by strapping or duct tape.
Note: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

Figure 1
Example of Cooler Label/Marking Locations



3.0 Packaging and Shipping Samples Preserved with Sodium Hydroxide

3.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Excepted Quantities of Sodium Hydroxide Preservatives

Preservative		***Desired in Final Sample***		***Quantity of Preservative (ml) for Specified Container***				
		pH	**Conc.**	**40 ml**	**125 ml**	**250 ml**	**500 ml**	**1 L**
NaOH	30%	>12	0.08%		.25	0.5	1	2

5 drops = 1 ml

3.2 Responsibility

It is the responsibility of the qualified shipper to determine the amount of preservative in each sample so that accurate determination of quantities can be made.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

3.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test
- Inner packings may consist of glass or plastic jars no larger than 1 pint
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

3.4 Packaging Samples Preserved with Sodium Hydroxide

Samples containing NaOH as a preservative that exceed the excepted concentration of 0.08 percent (2 ml of a 30 percent NaOH solution per liter) may be shipped as a limited quantity per packing instruction Y819 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited-quantity samples shipments:

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape before sampling.
- At a minimum the label must contain:
  - Project name
  - Project number
  - Date and time of sample collection
  - Sample location
  - Sample identification number
  - Collector's initials
  - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)
- This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
- Place glass containers inside a polyethylene bottle filled with vermiculite; seal the bottle.
- The total volume of sample in each cooler must not exceed 1 liter.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tieing or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

  Sodium Hydroxide Solution
  UN1824
  LTD. QTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.

Note: Samples meeting the exception concentration of 0.08 percent NaOH by weight may be shipped as nonregulated or nonhazardous following the procedure in Section 1.4.

Note: No marking or labeling can be obscured by strapping or duct tape.

Note: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

4.0 Packaging and Shipping Samples Preserved with Hydrochloric Acid

4.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Excepted Quantities of Hydrochloric Acid Preservatives

Preservative		***Desired in Final Sample***		***Quantity of Preservative (ml) for Specified Container***		
		pH	**Conc.**	**40 ml**	**125 ml**	**250 ml**
HCl	2N	<1.96	0.04%	.2	.5	1

5 drops = 1 ml

4.2 Responsibility

It is the responsibility of the qualified shipper to:

- Determine the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT
- Determine the amount of preservative in each sample so that accurate determination of quantities can be made

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

4.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3.

- Inner packing may consist of glass or plastic jars no larger than 1 pint.
- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test.
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

4.4 Packaging Samples Preserved with Hydrochloric Acid

The following steps are to be followed when packaging limited-quantity sample shipments:

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape before sampling.
- At a minimum the label must contain:
  - Project name
  - Project number
  - Date and time of sample collection
  - Sample location
  - Sample identification number
  - Collector's initials
  - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)
- Wrap each container (40-ml VOA vials) in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
- Place wrapped containers inside a polyethylene bottle filled with vermiculite; seal the bottle. (No more than 4 VOA vials will fit inside a 500-ml wide-mouth polyethylene bottle.)

- Total volume of sample inside each cooler must not exceed 1 liter.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tieing or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

  Hydrochloric Acid Solution
  UN1789
  LTD. QTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.

Note: Samples containing less than the exception concentration of 0.04 percent HCl by weight will be shipped as nonregulated or nonhazardous following the procedure in Section 1.4.

Note: No marking or labeling can be obscured by strapping or duct tape.

Note: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

5.0 Packaging and Shipping Samples Preserved with Nitric Acid

5.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Excepted Quantities of Nitric Acid Preservatives

Preservative		*Desired in Final Sample*		*Quantity of Preservative (ml) for Specified Container*				
		pH	**Conc.**	**40 ml**	**125 ml**	**250 ml**	**500 ml**	**1 L**
HNO_3	6N	<1.62	0.15%		2	4	5	8

5 drops = 1 mg/L

5.2 Responsibility

It is the responsibility of the qualified shipper to:

- Determine the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT
- Determine the amount of preservative in each sample so that accurate determination of quantities can be made

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

5.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Inner packings may consist of glass or plastic jars no larger than 100 ml.
- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test.
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

5.4 Packaging Samples Preserved with Nitric Acid

Samples containing HNO_3 as a preservative that exceed the excepted concentration of 0.15 percent HNO_3 will be shipped as a limited quantity per packing instruction Y807 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited-quantity sample shipments:

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape before sampling.
- At a minimum the label must contain:
  - Project name
  - Project number
  - Date and time of sample collection
  - Sample location
  - Sample identification number
  - Collector's initials
  - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)
- This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
- Place glass containers inside a polyethylene bottle filled with vermiculite; seal the bottle.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tieing or taping.
- The maximum volume of preserved solution in the cooler must not exceed 500 ml.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

Nitric Acid Solution (with less than 20 percent)
UN2031
Ltd. Qty.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.

Note: Samples meeting the exception concentration of 0.15 percent HNO_3 by weight will be shipped as nonregulated or nonhazardous following the procedure in Section 1.4.

Note: No marking or labeling can be obscured by strapping or duct tape.

Note: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

6.0 Packaging and Shipping Samples Preserved with Sulfuric Acid

6.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Excepted Quantities of Sulfuric Acid Preservatives

Preservative		***Desired in Final Sample***		***Quantity of Preservative (ml) for Specified Container***				
		pH	**Conc.**	**40 ml**	**125 ml**	**250 ml**	**500 ml**	**1 L**
H_2SO_4	37N	<1.15	0.35%	.1	.25	0.5	1	2

5 drops = 1 ml

6.2 Responsibility

It is the responsibility of the qualified shipper to:

- Determine the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT
- Determine the amount of preservative in each sample so that accurate determination of quantities can be made

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

6.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Inner packings may consist of glass or plastic jars no larger than 100 ml.
- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test.
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

6.4 Packaging of Samples Preserved with Sulfuric Acid

Samples containing H_2SO_4 as a preservative that exceed the excepted concentration of 0.35 percent will be shipped as a limited quantity per packing instruction Y809 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited-quantity samples shipments:

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape before sampling.
- At a minimum the label must contain:
  - Project name
  - Project number
  - Date and time of sample collection
  - Sample location
  - Sample identification number
  - Collector's initials
  - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)
- Wrap each glass container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
- Place glass containers inside a polyethylene bottle filled with vermiculite; seal the bottle.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tieing or taping.
- The maximum volume of preserved solution in the cooler must not exceed 500 ml.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

  Sulfuric Acid Solution
  UN2796
  LTD. QTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.

Note: Samples containing less than the exception concentration of 0.35 percent H_2SO_4 by weight will be shipped as nonregulated or nonhazardous in accordance with the procedure described in Section 1.4.

Note: No marking or labeling can be obscured by strapping or duct tape.

Note: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

7.0 Packaging and Shipping Limited-Quantity Radioactive Samples

7.1 Containers

The inner packaging containers that may be used for these shipments include:

- Any size sample container

7.2 Description/Responsibilities

- The qualified shipper will determine that the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT.
- The qualified shipper will ship all samples that meet the Class 7 definition of radioactive materials and meet the activity requirements specified in Table 7 of 49 CFR 173.425, as Radioactive Materials in Limited Quantity. The qualified shipper will verify that all packages and their contents meet the requirements of 49 CFR 173.421, *Limited Quantities of Radioactive Materials*.
- The packaging used for shipping will meet the general requirements for packaging and packages specified in 49 CFR 173.24 and the general design requirements provided in 173.410. These standards state that a package must be capable of withstanding the effects of any acceleration, vibration, or vibration resonance that may arise under normal condition of transport without any deterioration in the effectiveness of the closing devices on the various receptacles or in the integrity of the package as a whole and without loosening or unintentionally releasing the nuts, bolts, or other securing devices even after repeated use.
- If the shipment is from a DOE facility, radiological screenings will be completed on all samples taken. The qualified shipper will review the results of each screening (alpha, beta, and gamma speciation). Samples will not be shipped offsite until the radiological screening has been performed.
- The total activity for each package will not exceed the relevant limits listed in Table 7 of 49 CFR 173.425. The A_2 value of the material will be calculated based on all radionuclides found during previous investigations (if any) in the area from which the samples are derived. The A_2 values to be used will be the most restrictive of all potential radionuclides as listed in 49 CFR 173.435.
- The radiation level at any point on the external surface of the package bearing the sample(s) will not exceed 0.005 mSv/hour (0.5 mrem/hour). These will be verified by dose and activity monitoring before shipment of the package.
- The removable radioactive surface contamination on the external surface of the package will not exceed the limits specified in 49 CFR 173.443(a). CDM will apply the DOE-established free release criteria for removable surface contamination of less than 20 dpm/100 cm^2 (alpha) and 1,000 dpm/100 cm^2 (beta/gamma). It shall be noted that these values are more conservative than the DOT requirements for removable surface contamination.
- The qualified shipper will verify that the outside of the inner packaging is marked “Radioactive.”
- The qualified shipper will verify that the excepted packages prepared for shipment under the provisions of 49 CFR 173.421 have a notice enclosed, or shown on the outside of the package, that reads, **“This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910.”**

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

7.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Survey documentation/radiation screening results (if shipping from DOE or radiological sites)
- Orientation labels
- Excepted quantities label
- Consignor/consignee labels

7.4 Packaging of Limited-Quantity Radioactive Samples

The following steps are to be followed when packaging limited-quantity sample shipments:

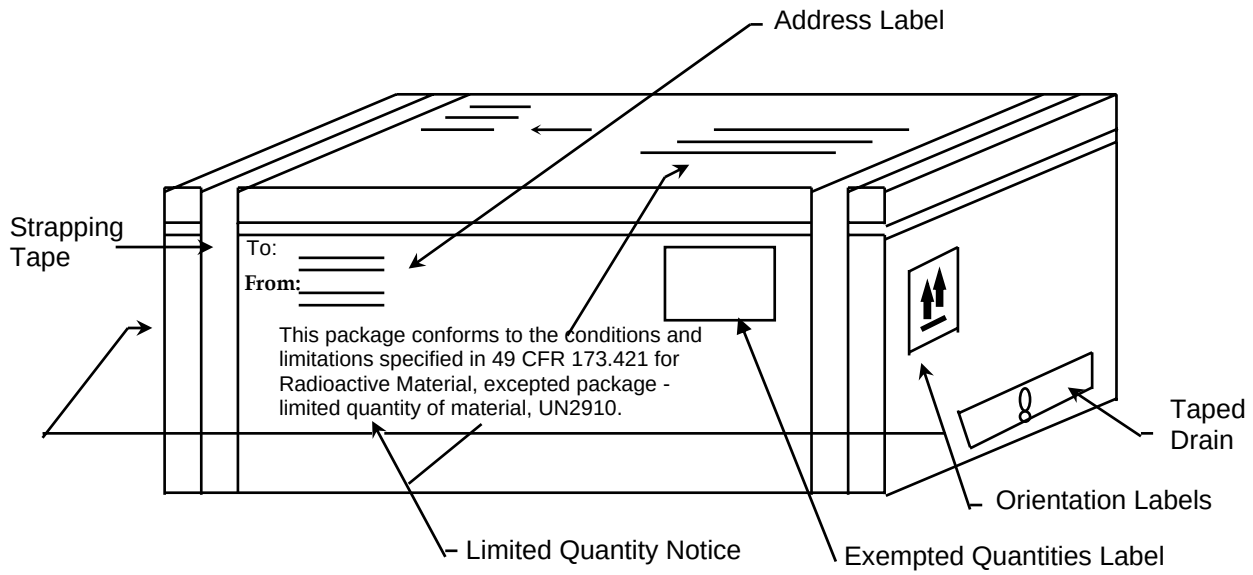
- The cooler is to be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape before sampling.
- At a minimum the label must contain:
  - Project name
  - Project number
  - Date and time of sample collection
  - Sample location
  - Sample identification number
  - Collector's initials
- This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place sufficient amount of vermiculite, or approved packaging material, in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- If required, place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tieing or taping.
- Place a label marked Radioactive on the outside of the sealed bag.
- Enclose a notice that includes the name of the consignor or consignee and the following statement: ***"This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910."***
- Note that both DOT and IATA apply different limits to the quantity in the inside packing and in the outside packing.
- The maximum weight of the package shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- If a cooler is used, wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix package orientation labels on two opposite sides of the cooler/package.
- Affix a completed Excepted Quantities label to the side of the cooler/package.
- Secure any marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of the cooler labeling/marking is shown in Figure 2.

Note: No marking or labeling can be obscured by strapping or duct tape.

- Complete the Shipment Quality Assurance Checklist (Appendix B).

Note: Except as provided in 49 CFR 173.426, the package will not contain more than 15 grams of ^{235}U.
Note: A declaration of dangerous goods is not required.

Figure 2
Radioactive Material – Limited-Quantity Cooler Marking Example



8.0 References

U. S. Environmental Protection Agency. Region IV. February 1991 or current. *Standard Operating Procedures and Quality Assurance Manual*.

_____. 1996 or current. *Sampler's Guide to the Contract Laboratory Program*, EPA/540/R-96/032.

Title 49 Code of Federal Regulations, Department of Transportation. 2005 or current revision. *Hazardous Materials Table, Special Provisions, Hazardous, Materials Communications, Emergency Response Information, and Training Requirements*, 49 CFR 172.

Title 49 Code of Federal Regulations, Department of Transportation. 2005 or current revision. *Shippers General Requirements for Shipments and Packagings*, 49 CFR 173.

Appendix A

Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity

Sample Packaging

Yes	No	N/A	
❑	❑	❑	The VOA vials are wrapped in bubble wrap and placed inside a zip-type bag.
❑	❑	❑	The VOA vials are placed into a polyethylene bottle, filled with vermiculite, and tightly sealed.
❑	❑	❑	The drain plug is taped inside and outside to ensure control of interior contents.
❑	❑	❑	The samples have been placed inside garbage bags with sufficient bags of ice to preserve samples at 4°C.
❑	❑	❑	The cooler weighs less than the 66-pound limit for limited-quantity shipment.
❑	❑	❑	The garbage bag has been sealed with tape (or tied) to prevent movement during shipment.
❑	❑	❑	The chain-of-custody has been secured to the interior of the cooler lid.
❑	❑	❑	The cooler lid and sides have been taped to ensure a seal.
❑	❑	❑	The custody seals have been placed on both the front and back hinges of the cooler, using waterproof tape.

Air Waybill Completion

Yes	No	N/A	
❑	❑	❑	Section 1 has the shipper's name, company, and address; the account number, date, internal billing reference number; and the telephone number where the shipper can be reached.
❑	❑	❑	Section 2 has the recipient's name and company along with a telephone number where they can be reached.
❑	❑	❑	Section 3 has the **Bill Sender** box checked.
❑	❑	❑	Section 4 has the **Standard Overnight** box checked.
❑	❑	❑	Section 5 has the **Deliver Weekday** box checked.
❑	❑	❑	Section 6 has the number of packages and their weights filled out. Was the total of all packages and their weights figured up and added at the bottom of Section 6?
❑	❑	❑	Under the **Transport Details** box, the **Cargo Aircraft Only** box is obliterated, leaving only the **Passenger and Cargo Aircraft** box.
❑	❑	❑	Under the **Shipment Type**, the **Radioactive** box is obliterated, leaving only the **Non-Radioactive** box.
❑	❑	❑	Under the **Nature and Quantity of Dangerous Goods** box, the **Proper Shipping Name, Class or Division, UN or ID No., Packing Group, Subsidiary Risk, Quantity and Type of Packing, Packing Instructions, and Authorization** have been filled out for the type of chemical being sent.
❑	❑	❑	The **Name, Place and Date, Signature, and Emergency Telephone Number** appears at the bottom of the FedEx Airbill.
❑	❑	❑	The statement "In accordance with IATA/ICAO" appears in the **Additional Handling Information** box.
❑	❑	❑	The E**mergency Contact Information** at the bottom of the FedEx Airbill is truly someone who can respond any time of the day or night.

Proper Shipping Name	*Class or Division*	*UN or ID No.*	*Packing Group*	*Sub Risk*	*Quantity*	*Packing Instruction*	*Authorization*
Hydrochloric Acid Solution	8	UN1789	II		1 plastic box × 0.5 L	Y809	Ltd. Qty.
Nitric Acid Solution (with less than 20%)	8	UN2031	II		1 plastic box × 0.5 L	Y807	Ltd. Qty.
Sodium Hydroxide Solution	8	UN1824	II		1 plastic box × 0.5 L	Y809	Ltd. Qty.
Sulfuric Acid Solution	8	UN2796	II		1 plastic box × 0.5 L	Y809	Ltd. Qty.
Methanol	3	UN1230	II		1 plastic box × 1 L	Y305	Ltd. Qty.

Sample Cooler Labeling

Yes	No	N/A	
❑	❑	❑	The proper shipping name, UN number, and Ltd. Qty. appears on the shipping container.
❑	❑	❑	The corresponding hazard labels are affixed on the shipping container; the labels are not obscured by tape.
❑	❑	❑	The name and address of the shipper and receiver appear on the top and side of the shipping container.
❑	❑	❑	The air waybill is attached to the top of the shipping container.
❑	❑	❑	**Up Arrows** have been attached to opposite sides of the shipping container.
❑	❑	❑	Packaging tape does not obscure markings or labeling.

Appendix B
Shipment Quality Assurance Checklist

Date: _____ Shipper: _____ Destination: _____

Item(s) Description: _____

Radionuclide(s): _____

Radiological Survey Results: surface _____mrem/hr 1 meter _____

Instrument Used: Mfgr: _____ Model: _____

S/N: _____ Cal Date: _____

Limited-Quantity or Instrument and Article

Yes	No		
___	___	1.	Strong tight package (package that will not leak material during conditions normally incidental to transportation).
___	___	2.	Radiation levels at any point on the external surface of package less than or equal to 0.5 mrem/hr.
___	___	3.	Removable surface contamination less than 20 dpm/100 cm^2 (alpha) and 1,000 dpm/100 cm^2 (beta/gamma).
___	___	4.	Outside inner package bears the marking "Radioactive."
___	___	5.	Package contains less than 15 grams of ^{235}U (check yes if ^{235}U not present).
___	___	6.	Notice enclosed in or on the package that includes the consignor or consignee and the statement, **"This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910."**
___	___	7.	Activity less than that specified in 49 CFR 173.425. Permissible package limit: Package Quantity:
___	___	8.	On all air shipments, the statement **Radioactive Material, excepted package-limited quantity of material** shall be noted on the air waybill.

Qualified Shipper: _____ Signature: _____

Attachment E

SOP 4-1 Field Logbook Content Control

Draft

Field Logbook Content and Control

SOP 4-1
Revision: 6
Date: March 2007

Prepared: Del Baird

Technical Review: Laura Splichal

E-Signed by Michael C. Malloy
VERIFY authenticity with ApproveIt

QA Review: Jo Nell Mullins

Approved:

Signature/Date

E-Signed by P. Michael Schwan
VERIFY authenticity with ApproveIt

Issued:

Signature/Date

1.0 Objective

The objective of this standard operating procedure (SOP) is to set CDM Federal (CDM) criteria for content entry and form of field logbooks. Field logbooks are an essential tool to document field activities for historical and legal purposes.

2.0 Background

2.1 Definitions

Biota - The flora and fauna of a region.

Magnetic Declination Corrections - Compass adjustments to correct for the angle between magnetic north and geographical meridians.

2.2 Discussion

Information recorded in field logbooks includes field team names; observations; data; calculations; date/time; weather; and description of the data collection activity, methods, instruments, and results. Additionally, the logbook may contain deviations from plans and descriptions of wastes, biota, geologic material, and site features including sketches, maps, or drawings as appropriate.

3.0 General Responsibilities

Field Team Leader (FTL) - The FTL is responsible for ensuring that the format and content of data entries are in accordance with this procedure.

Site Personnel - All CDM employees who make entries in field logbooks during onsite activities are required to read this procedure before engaging in this activity. The FTL will assign field logbooks to site personnel who will be responsible for their care and maintenance. Site personnel will return field logbooks to the records file at the end of the assignment.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities should be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Required Equipment

- Site-specific plans
- Field logbook
- Indelible black or blue ink pen
- Ruler or similar scale

5.0 Procedures

5.1 Preparation

In addition to this SOP, site personnel responsible for maintaining logbooks must be familiar with all procedures applicable to the field activity being performed. These procedures should be consulted as necessary to obtain specific information about equipment and supplies, health and safety, sample collection, packaging, decontamination, and documentation. These procedures should be located at the field office or vehicle for easy reference.

Field logbooks shall be bound with lined, consecutively numbered pages. All pages must be numbered before initial use of the logbook. Before use in the field, each logbook will be marked with a specific document control number issued by

the document control administrator, if required by the contract quality implementation plan (QIP). Not all contracts require document control numbers. The following information shall be recorded on the cover of the logbook:

- Field logbook document control number (if applicable).
- Activity (if the logbook is to be activity-specific), site name, and location.
- Name of CDM contact and phone number(s) (typically the project manager).
- Start date of entries.
- End date of entries.
- In specific cases, special logbooks may be required (e.g., waterproof paper for stormwater monitoring).

The first few (approximately five) pages of the logbook will be reserved for a table of contents (TOC). Mark the first page with the heading and enter the following:

Table of Contents

Date/Description	Pages
(Start Date)/Reserved for TOC	1-5

The remaining pages of the table of contents will be designated as such with "TOC" written on the top center of each page. The table of contents should be completed as activities are completed and before placing the logbook in the records file.

5.2 Operation

Requirements that must be followed when using a logbook:

- Record work, observations, quantities of materials, calculations, drawings, and related information directly in the logbook. If data collection forms are specified by an activity-specific plan, this information does not need to be duplicated in the logbook. However, any forms used to record site information must be referenced in the logbook.
- Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made. Use both sides of each page.
- Do not erase or blot out any entry at any time. Indicate any deletion by a single line through the material to be deleted. Initial and date each deletion. Take care to not obliterate what was written previously.
- Do not remove any pages from the book.

Specific requirements for field logbook entries include:

- Initial and date each page.
- Sign and date the final page of entries for each day.
- Initial and date all changes.
- Multiple authors must sign out the logbook by inserting the following:
  Above notes authored by:
  - (Sign name)
  - (Print name)
  - (Date)
- A new author must sign and print his/her name before additional entries are made.
- Draw a diagonal line through the remainder of the final page at the end of the day.
- Record the following information on a daily basis:
  - Date and time
  - Name of individual making entry
  - Names of field team and other persons onsite
  - Description of activity being conducted including station or location (i.e., well, boring, sampling location number) if appropriate
  - Weather conditions (i.e., temperature, cloud cover, precipitation, wind direction, and speed) and other pertinent data
  - Level of personal protection used
  - Serial numbers of instruments
  - Equipment calibration information
  - Serial/tracking numbers on documentation (e.g., carrier air bills)

Entries into the field logbook shall be preceded with the time (written in military units) of the observation. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged. All measurements made and samples collected must be recorded unless they are documented by automatic methods (e.g., data logger) or on a separate form required by an operating procedure. In these cases, the logbook must reference the automatic data record or form.

At each station where a sample is collected or an observation or measurement made, a detailed description of the location of the station is required. Use a compass (include a reference to magnetic declination corrections), scale, or nearby survey markers, as appropriate. A sketch of station location may be warranted. All maps or sketches made in the logbook should have descriptions of the features shown and a direction indicator. It is preferred that maps and sketches be oriented so that north is toward the top of the page. Maps, sketches, figures, or data that will not fit on a logbook page should be referenced and attached to the logbook to prevent separation.

Other events and observations that should be recorded include:

- Changes in weather that impact field activities.
- Deviations from procedures outlined in any governing documents. Also record the reason for any noted deviation.
- Problems, downtime, or delays.
- Upgrade or downgrade of personal protection equipment.
- Visitors to the site.

5.3 Post-Operation

To guard against loss of data as a result of damage or disappearance of logbooks, completed pages shall be periodically photocopied (weekly, at a minimum) and forwarded to the field or project office. Other field records shall be photocopied and submitted regularly and as promptly as possible to the office. When possible, electronic media such as disks and tapes should be copied and forwarded to the project office.

At the conclusion of each activity or phase of site work, the individual responsible for the logbook will ensure that all entries have been appropriately signed and dated and that corrections were made properly (single lines drawn through incorrect information, then initialed and dated). The completed logbook shall be submitted to the records file.

6.0 Restrictions/Limitations

Field logbooks constitute the official record of onsite technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by CDM personnel and their subcontractors. They are documents that may be used in court to indicate dates, personnel, procedures, and techniques employed during site activities. Entries made in these logbooks should be factual, clear, precise, and nonsubjective. Field logbooks, and entries within, are not to be used for personal use.

7.0 References

Sandia National Laboratories. 1991. *Procedure for Preparing Sampling and Analysis Plan, Site-Specific Sampling Plan, and Field Operating Procedures,* QA-02-03. Albuquerque Environmental Program, Department 3220, Albuquerque, New Mexico.

Sandia National Laboratories. 1992. *Field Operation Procedure for Field Logbook Content and Control*. Environmental Restoration Department, Division 7723, Albuquerque, New Mexico.

Photographic Documentation of Field Activities

SOP 4-2
Revision: 7
Date: March 2007

Prepared: David O. Johnson

Technical Review: Sharon Budney

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VERIFY authenticity with ApproveIt

QA Review: Jo Nell Mullins

Approved:

Signature/Date

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Issued:

Signature/Date

1.0 Objective

The purpose of this standard operating procedure (SOP) is to provide standard guidelines and methods for photographic documentation, which include still and digital photography and videotape or DVD recordings of field activities and site features (geologic formations, core sections, lithologic samples, water samples, general site layout, etc.). This document shall provide guidelines designed for use by a professional or amateur photographer. This SOP is intended for circumstances when formal photographic documentation is required. Based on project requirements, it may not be applicable for all photographic activities.

2.0 Background

2.1 Definitions

Photographer - A photographer is the camera operator (professional or amateur) of still photography, including digital photography, or videotape or digital versatile discs (DVD) recording whose primary function with regard to this SOP is to produce documentary or data-oriented visual media.

Identifier Component - Identifier components are visual components used within a photograph such as visual slates, reference markers, and pointers.

Standard Reference Marker - A standard reference marker is a reference marker that is used to indicate a feature size in the photograph and is a standard length of measure, such as a ruler, meter stick, etc. In limited instances, if a ruled marker is not available or its use is not feasible, it can be a common object of known size placed within the visual field and used for scale.

Slates - Slates are blank white index cards or paper used to present information pertaining to the subject/procedure being photographed. Letters and numbers on the slate will be bold and written with black indelible marking pens.

Arrows and Pointers - Arrows and pointers are markers/pointers used to indicate and/or draw attention to a special feature within the photograph.

Contrasting Backgrounds - Contrasting backgrounds are backdrops used to lay soil samples, cores, or other objects on for clearer viewing and to delineate features.

Data Recording Camera Back - A data recording camera back is a camera attachment or built-in feature that will record, at the very least, frame numbers and dates directly on the film.

2.2 Associated Procedures

- CDM Federal SOP 4-1, *Field Logbook Content and Control*

2.3 Discussion

Photographs and videotape or DVD recordings made during field investigations are used as an aid in documenting and describing site features, sample collection activities, equipment used, and possible lithologic interpretation. This SOP is designed to illustrate the format and desired placement of identifier components, such as visual slates, standard

reference markers, and pointers. These items shall become an integral part of the "visual media" that, for the purpose of this document, shall encompass still photographs, digital photographs, videotape recordings (or video footage), and recordings on DVDs. The use of a photographic logbook and standardized entry procedures are also outlined. These procedures and guidelines will minimize potential ambiguities that may arise when viewing the visual media and ensure the representative nature of the photographic documentation.

3.0 General Responsibilities

Field Team Leader - The field team leader (FTL) is responsible for ensuring that the format and content of photographic documentation are in accordance with this procedure. The FTL is responsible for directing the photographer to specific situations, site features, or operations that the photographer will be responsible for documenting.

Photographer - The photographer shall seek direction from the FTL and regularly discuss the visual documentation requirements and schedule. The photographer is responsible for maintaining a logbook per Sections 5.1, 5.2.4, and 5.3.1 of this SOP. Responsibilities will be defined in the project sampling plan.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

4.0 Required Equipment

A general list of equipment that may be used:

- 35mm camera or disposable single use camera (35mm or panoramic use)
- Digital camera
- Extra batteries for 35mm camera
- Video camera and appropriate storage media (e.g., video tapes, DVDs)
- Logbook
- Indelible black or blue ink pen
- Standard reference markers
- Slates
- Arrows or pointers
- Contrasting backgrounds
- Medium speed, or multi purpose fine-grain, color, 35mm negative film or slide film (project dependent)
- Data recording camera back (if available)
- Storage medium for digital camera

5.0 Procedures

5.1 Documentation

A commercially available, bound logbook will be used to log and document photographic activities. Review CDM Federal SOP 4-1, *Field Logbook Content and Control* and prepare all supplies needed for logbook entries.

Note: A separate photographic logbook is not required. A portion of the field logbook may be designated as the photographic log and documentation section.

Field Health and Safety Considerations

There are no hazards that an individual will be exposed to specific to photographic documentation. However, site-specific hazards may arise depending on location or operation. Personal protective equipment used in this operation will be site-specific and dictated through requirements set by the site safety officer, site health and safety plan, and/or prescribed by the CDM Federal Corporate Health and Safety Program. The photographer should contact the site safety officer for health and safety orientation before commencing field activities. The site health and safety plan must be read before entry to the site, and all individuals must sign the appropriate acknowledgement that this has been done.

The photographer should be aware of any potential physical hazards while photographing the subject (e.g., traffic, low overhead hazard, edge of excavation).

5.2 Operation

5.2.1 General Photographic Activities in the Field

The following sections provide general guidelines that should be followed to visually document field activities and site features using still/digital cameras and video equipment. Listed below are general suggestions that the photographer should consider when performing activities under this SOP:

- The photographer should be prepared to make a variety of shots, from close-up to wide-angle. Many shots will be repetitive in nature or format, especially close-up site feature photographs. Consideration should therefore be given to designing a system or technique that will provide a reliable repetition of performance.
- All still film photographs should be made using a medium speed, or multi purpose fine-grain, color negative film in the 35mm format unless otherwise directed by the FTL.
- It is suggested that Kodak brand "Ektapress Gold Deluxe" film or equivalent be used as the standard film for the still photography requirements of the field activities. This film is stable at room temperature after exposure and will better survive the time lag between exposure and processing. It is suggested that film speed ASA 100 should be used for outdoor photographs in bright sunlight, ASA 200 film should be used in cloudy conditions, and ASA 400 film should be used indoors or for very low-light outdoor photographs.
- No preference of videotape or DVD brand along with digital storage medium is specified and is left to the discretion of the photographer.
- The lighting for sample and feature photography should be oriented toward a flat condition with little or no shadow. If the ambient lighting conditions are inadequate, the photographer should be prepared to augment the light (perhaps with reflectors or electronic flash) to maintain the desired visual effect.
- Digital cameras have multiple photographic quality settings. A camera that obtains a higher resolution (quality) has a higher number of pixels and will store a fewer number of photographs per digital storage medium.

5.2.2 General Guidelines for Still Photography

Slate Information

It is recommended that each new roll of film or digital storage medium shall contain on the first usable frame (for film) a slate with consecutively assigned control numbers (a consecutive, unique number that is assigned by the photographer as in sample numbers).

Caption Information

All still photographs will have a full caption permanently attached to the back or permanently attached to a photo log sheet. The caption should contain the following information (digital photographs should have a caption added after the photographs are downloaded):

- Film roll control number (if required) and photograph sequence number
- Date and time
- Photographer
- Description of activity/item shown (e.g., name of facility/site, specific project name, project number)
- Direction (if applicable)

When directed by the sampling plan, a standard reference marker should be used in all documentary visual media. While the standard reference marker will be predominantly used in close-up feature documentation, inclusion in all scenes should be considered.

Digital media should be downloaded at least once each day to a personal computer; the files should be in either "JPEG" or "TIFF" format. Files should be renamed at the time of download to correspond to the logbook. It is recommended the electronic files be copied to a compact disc for backup.

Close-Up and Feature Photography

When directed by the sampling plan, close-up photographs should include a standard reference marker of appropriate size as an indication of the feature size and contain a slate marked with the site name and any identifying label, such as a well number or core depth, that clearly communicates to the viewer the specific feature being photographed.

Feature samples, core pieces, and other lithologic media should be photographed as soon as possible after they have been removed from their in situ locations. This enables a more accurate record of their initial condition and color. When directed by the sampling plan, include a standard reference color strip (color chart such as Munsell Soil Color Chart or that available from Eastman Kodak Co.) within the scene. This is to be included for the benefit of the viewer of the photographic document and serves as a reference aid to the viewer for formal lithologic observations and interpretations.

Site Photography
Site photography, in general, will consist predominantly of medium- and wide-angle shots. A standard reference marker should be placed adjacent to the feature or, when this is not possible, within the same focal plane.

While it is encouraged that a standard reference marker and caption/slate be included in the scene, it is understood that situations will arise that preclude their inclusion within the scene. This will be especially true of wide-angle shots. In such a case, the film/tape control number shall be entered in the photographic logbook along with the frame number and all other information pertinent to the scene.

Panoramic
In situations where a wide-angle lens does not provide sufficient subject detail, a single-use disposable panoramic camera is recommended. If this type of camera is not available, a panoramic series of two or three photos would be appropriate. Panoramas can provide greater detail while covering a wide subject, such as an overall shot of a site.

To shoot a panoramic series using a standard 35mm or digital camera, the following procedures are recommended:

- Use a stable surface or tripod to support the camera
- Allow a 20- to 30-percent overlap while maintaining a uniform horizon
- Complete two to three photos per series

5.2.3 General Photographic Documentation Using Video Cameras
As a reminder, it is not within the scope of this document to set appropriate guidelines for presentation or “show” videotape or DVD recording. The following guidelines are set for documentary videotape or DVD recordings only and should be implemented at the discretion of the site personnel.

Documentary videotape or DVD recordings of field activities may include an audio slate for all scenes. At the beginning of each video session, an announcer will recite the following information: date, time (in military units), photographer, site ID number, and site location. This oral account may include any additional information clarifying the subject matter being recorded.

A standard reference marker may be used when taking close-up shots of site features with a video camera. The scene may also include a caption/slate. It should be placed adjacent and parallel to the feature being photographed.

It is recommended that a standard reference marker and caption/slate be included in all scenes. The caption information is vital to the value of the documentary visual media and should be included. If it is not included within the scene, it should be placed before the scene.

Original video recordings will not be edited. This will maintain the integrity of the information contained on the videotape or DVD. If editing is desired, a working copy of the original video recording can be made.

A label should be placed on the videotape or DVD with the appropriate identifying information (project name, project number, date, location, etc.).

5.2.4 Photographic Documentation
Photographic activities must be documented in a photographic logbook or in a section of the field logbook. The photographer will be responsible for making proper entries.

In addition to following the technical standards for logbook entry as referenced in CDM Federal SOP 4-1, the following information should be maintained in the appropriate logbook:

- Photographer name.
- If required, an entry shall be made for each new roll/tape/DVD control number assigned.
- Sequential tracking number for each photograph taken (for digital cameras, the camera-generated number may be used).
- Date and time (military time).
- Location.
- A description of the activity/item photographed.
- If needed, a description of the general setup, including approximate distance between the camera and the subject, may be recorded in the logbook.
- Record as much other information as possible to assist in the identification of the photographic document.

5.3 Post Operation

All film will be sent for development and printing to a photographic laboratory (to be determined by the photographer). The photographer will be responsible for arranging transport of the film from the field to the photographic laboratory. The photographer shall also be responsible for arranging delivery of the negatives and photographs, digital storage medium, or videotape or DVD to the project management representative to be placed in the project files.

5.3.1 Documentation

At the end of each day's photographic session, the photographer(s) will ensure that the appropriate logbook has been completely filled out and maintained as outlined in CDM Federal SOP 4-1.

5.3.2 Archive Procedures

- Photographs and the associated set of uncut negatives, digital media, and original unedited documentary video recordings will be submitted to the project files and handled according to contract records requirements. The project manager will ensure their proper distribution.
- Completed pages of the appropriate logbook will be copied weekly and submitted to the project files.

6.0 Restrictions/Limitations

This document is designed to provide a set of guidelines for the field amateur or professional photographer to ensure that an effective and standardized program of visual documentation is maintained.

It is not within the scope of this document to provide instruction in photographic procedures, nor is it within the scope of this document to set guidelines for presentation or "show" photography.

The procedures outlined herein are general by nature. The photographer is responsible for specific operational activity or procedure. Questions concerning specific procedures or requirements should be directed to the project manager or FTL.

Note: Some sites do not permit photographic documentation. Check with the site contact for any restrictions.

7.0 References

U. S. Army Corps of Engineers. 2001. *Requirements for the Preparation of Sampling and Analysis Plans*, EM 200-1-3. Appendix F. February.

U. S. Environmental Protection Agency. 1992. National Enforcement Investigations Center. *Multi-Media Investigation Manual*, EPA-330/9-89-003-R. p. 85. Revised March.

_____. Region IV. 2001. *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*. Athens, Georgia. November.

NORTHEAST ANALYTICAL INC.

LABORATORY QUALITY ASSURANCE PLAN

November 12, 2007

2190 Technology Drive Schenectady, NY 12308
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LABORATORY QUALITY ASSURANCE PLAN

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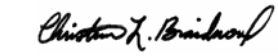
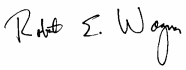


Prepared by:_____ Date: 11/12/2007
Christina Braidwood- Quality Assurance Officer



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Semi-Volatile Organics Section Supervisor-Roy Smith	07	10/3/07
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Extractions Department Manager-Michael P. Glenn	09	10/3/07
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QUALITY ASSURANCE PLAN

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QUALITY ASSURANCE PLAN

REVISION INFORMATION

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QAP_04	09	12-November-2007	Christina L. Braidwood, QA Officer Robert E. Wagner, Laboratory Director
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QAP_04	05	5-October-2006	William A. Kotas, QA Officer Robert E. Wagner, Laboratory Director
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QAP_04	02	22-July-2002	William A. Kotas, QA Officer Robert E. Wagner, Laboratory Director
QAP_04	01	14-April-2000	William A. Kotas, QA Officer Robert E. Wagner, Laboratory Director
QAP_03	04	24-December-1999	William A. Kotas, QA Officer Robert E. Wagner, Laboratory Director

1.0 QUALITY ASSURANCE INTRODUCTION

1.1 GENERAL

Northeast Analytical, Inc. (NEA), is committed to the generation of the highest quality data. This is achieved by employing state-of-the-art instrumentation, the latest validated methods and highly qualified staff. To ensure accountability, sample integrity and data accuracy this quality assurance (QA) program has been implemented. NEA recognizes that analytical qualitative and quantitative results are the key to the decision making process. We also understand that incorrect or inaccurate results may lead to faulty interpretations or poor decisions that would be worse than no results at all.

Northeast Analytical Inc. prides itself on quality work and client satisfaction. NEA's spectrum of clients has ranged from individual homeowners to some of the largest national and international organizations. References from individual clients are available upon request.

1.2 QUALITY ASSURANCE PROGRAM

Northeast Analytical is approved by the New York State Department of Health Environmental Laboratory Approval Program (ELAP), which is a Primary Accrediting Authority for the National Environmental Laboratory Approval Program (NELAP). Procedures used at the laboratory are designed to meet or exceed quality assurance guidelines established by ELAP, NELAP, and the US-EPA. This document has been designed to describe the QA program at NEA. The following procedures allow the laboratory to assess analytical performance, validate standard operating procedures and insure that accuracy and precision goals are being met. Detailed descriptions of analytical methods and procedures can be found in the Standard Operation Procedures (SOP) for the individual methods.

1.3 ANALYTICAL METHODS

When applicable NEA uses published methods from the EPA, New York State Department of Environmental Conservation (NYS-DEC), ASTM, AOAC, Standard Methods, NIOSH, and other sources. Custom analytical services are also available to meet client's unique requirements. All laboratory methods are outlined and documented in SOP documents. The SOP contains detailed descriptions of the laboratory procedures including modifications to published procedures. Since SOPs are used to document the current and historical procedures followed to analyze a sample, all versions and revisions are permanently archived.

1.4 LABORATORY FACILITIES AND SERVICES

Northeast Analytical, Inc. is located in a modern 15,000 sq. ft. facility in Riverside Technology Park in the city of Schenectady, New York. The laboratory building constructed in 1999 has doubled our previous laboratory space and provides room for expansion of services. The facility is centrally located in upstate New York, in close proximity to the cities of Albany and Troy. It is approximately three hours north of New York City, three hours west of Boston and two hours east of Syracuse NY.

Address: Northeast Analytical, Inc.
2190 Technology Drive
Riverside Technology Park
Schenectady, New York 12308
Phone: (518) 346 - 4592
FAX: (518) 381 - 6055
e-Mail: information@nealab.com

The facility houses the following laboratory and administrative units:

Non-Volatile Organic Extraction Lab
GC and GC-MS Instrument Analysis Lab
Trace Level Aqueous Organics Extraction Lab.
Volatile Organics Analysis Lab

Sample Cooler/Freezer Storage Area
Inorganics Analysis Lab
Inorganics /TCLP Preparation Lab
Dishwashing Facility
Sample Login Area
Sample Container Storage Room
Waste Storage Room
Data Archive/Storage Room
Computer Network Center
Administrative Offices

Additionally Northeast Analytical, Inc. has the following physical services:

- 10' x 14' walk-in sample cooler
- 12' x 17' walk-in sample freezer
- Ultra pure 18 mega ohm water system
- 12 individual laboratory hoods for samples and standard preparation
- 1 - 1992 Chevrolet 1/2 ton 4 x 4 pickup truck
- 1 - 2002 Toyota 2 x 2 pickup truck

2.0 QUALITY ASSURANCE POLICY STATEMENTS

2.1 QA POLICY

Northeast Analytical continuously strives to produce the highest quality data and reports. NEA adheres to the strict QA/QC program outlined in the NYS ELAP/NELAP manuals. Northeast Analytical Inc. warrants that it will employ professionally qualified personnel to test samples using, whenever possible, established and generally accepted and/or certified test methods. Northeast Analytical is committed to routinely performing laboratory work in compliance with the NELAC standard adopted by New York State ELAP.

Northeast Analytical, Inc. makes no other warranties, expressed or implied.

2.2 CONFIDENTIALITY

All sample, client and results information is deemed confidential between Northeast Analytical, Inc. and the client. All laboratory employees are required to sign confidentiality statements and instructed not to discuss client matters with anyone other than the client. Also NEA has all visitors that enter into laboratory areas in which client information may be seen sign a confidentiality agreement. A copy of the confidentiality statement signed by all employees and/or visitors is shown in Appendix A. NEA requires written requests from the client to transfer data to other parties.

2.3 ETHICAL AND LEGAL RESPONSIBILITIES

Northeast Analytical Inc. promotes strict adherence to requirements of promulgated test methods and approved quality plans. Any deliberate falsification of test results or deliberate misapplication of test procedures is grounds for immediate dismissal. All employees are required to sign an affidavit stating that they are responsible for conducting their work in an ethical manner when they are hired. Northeast Analytical has developed a proactive program for detecting fraud that includes periodic internal training seminars for employees, which describes ethical responsibilities and potential penalties for improper, unethical, or illegal actions.

2.4 COMPLAINTS POLICY

Client satisfaction is held as the key to success of our commercial laboratory. All complaints are handled in a timely fashion. Serious complaints are recorded using conversation/transmittal logs. Complaints are maintained in a NELAP Specified Complaints Log File. Investigation of a complaint may warrant issuance of a corrective action report (CAR) described in section 10.5. (See also section 10.6.4) The results an investigation and corrective action performed are maintained in the complaints log.

3.0 ORGANIZATIONAL STRUCTURE AND RESPONSIBILITIES

3.1 INTRODUCTION

Northeast Analytical's primary function is to provide high quality analytical testing services. To meet and maintain quality services, NEA employs qualified scientists and technicians well versed in analytical and environmental chemistry. The expertise and experience of the management and senior chemists plays an important role in project supervision and the training of laboratory technicians to perform chemical analyses correctly and produce highly respected analytical information.

NEA also believes that the training and caliber of the staff is directly reflected in the quality of work delivered, which is why the laboratory promotes internal seminars, presentations, and training in addition to external training and conferences organized by vendors, professional groups, and regulatory agencies.

3.2 LABORATORY ORGANIZATION STRUCTURE

Northeast Analytical, Inc. is a privately owned company, which is owned and operated by Robert E. Wagner and Robert W. Stoll. The laboratory is organized into four basic departments; Organics, Inorganics, Office/Project Management and Quality Assurance / Quality Control (QAU). The organization structure is outline in the flow chart in Appendix B.

3.3 LABORATORY STAFF

Statement of qualifications and resumes for Northeast Analytical's key staff are presented in Appendix C of this manual.

3.4 TRAINING

Each employee of NEA is issued a Personnel Training Log which documents specific training received at Northeast Analytical Inc. All training is conducted by qualified instructors. When the specific training is completed the trainee receives approval from the instructor that is documented in his/her training log. The trainee may at the judgment of the instructor be approved to perform the given task or both perform and instruct in the given task. An example page from the employee-training log is included in Appendix D of this document. An analyst or technician is required to perform and initial Demonstration of Competency, which includes successful analysis of method precision and accuracy samples before the analyst may work unsupervised on client samples. An Initial Demonstration of Competency Certificate is maintained in the employee’s personnel file and training records file.

3.5 RELATIONSHIP OF MANAGEMENT AND QUALITY ASSURANCE UNIT

The Quality Assurance Unit functions as an independent entity from laboratory technical operations and unit managers. The Quality Assurance Unit (QAU) is administered by the Quality Assurance Officer. The Quality Assurance Officer reports directly to the Laboratory Director. The Laboratory Director is responsible for ensuring that the functions of the Quality Assurance Unit are discharged properly, that the Quality Assurance Plan (QAP) is communicated to all appropriate personnel, and that the principles and requirements QAP are adhered to. The Laboratory Director is also responsible for ensuring that personnel meet the appropriate educational and/or technical requirements for their job function.

4.0 SAMPLE CUSTODY, STORAGE AND TRACKING

4.1 INTRODUCTION

All samples at Northeast Analytical Inc. are received, stored and tracked in accordance with New York State Environmental Laboratory Approval Program (ELAP) and National Environmental Laboratory Approval Program (NELAP) requirements. Further details regarding sample management is outlined in the Sample Login Management SOP document.

4.2 SAMPLE TRACKING, LOGIN AND IDENTIFICATION

Samples are tracked internally using the NEA Laboratory Information Management System (LIMS) computer program. The NEA_LIMS assigns unique sample ID numbers and records storage locations for samples.

All sample custody transfers between NEA and clients or subcontracting organizations are recorded on the sample chain-of-custody (COC). The COC document provides detail about the custody transfer including date/time sampled and received matrix type, sample condition, preservative, storage temperature, client identification, NEA identification number and required analyses. An example of an NEA chain-of-custody document is included in Appendix E of this manual.

4.3 SAMPLE STORAGE

Samples are stored according to NYS ELAP and US-EPA method requirements.

4.3.1 SAMPLE INTEGRITY AND CONTAMINATION

Samples are segregated according to storage requirements for the method and to minimize possible cross-sample contamination (ie: volatile samples are stored independent from other samples). NEA also maintains a sample storage blank quality assurance program to ensure that samples are not contaminated through laboratory storage.

4.3.2 SAMPLE PRESERVATION AND HOLD TIMES

Sample preservation and extraction/analytical hold time are noted and tracked using NEA_LIMS "Sample Progress Folders". NEA strives to inform clients of any deviations or possible problems with by using either NYS ELAP/NELAP or US-EPA holding time and preservative requirements upon sample receipt. In the event that a sample is received past extraction holding time or with insufficient hold time remaining to extract or analyze the sample the client will be notified and given the choice to proceed with sample analysis. All deviations from regulatory requirements will be noted on the certificate of analysis. Northeast Analytical provides the required preservative agent in sample containers as per request of the client. In cases where the client adds preservative to the samples in the field the client is asked to fill out the chain of custody indicating the preservative added. If no preservative is indicated Northeast Analytical will add the required preservative upon receipt of the sample.

For additional information regarding sample preservation and holding requirements please consult The **Guide to Environmental Analytical Methods** 4th Ed. (Edited by NEA Staff) available from Genium Publishing Corporation, 1171 Riverfront Center Amsterdam, NY 12010 (518) 842-4111.

4.3.3 SAMPLE AND EXTRACT ARCHIVING

NEA's policy is to maintain remaining raw samples for greater than 90 days after sample receipt and to hold the sample extracts for at least 90 days after sample analysis. Long-term archive facilities are available at NEA for an additional fee.

4.4 INTERNAL SAMPLE CUSTODY

Custody of samples is maintained in secure storage areas. NEA maintains individual "Sample LRF Folders" for each group of samples. The folder tracks sample progress and contains raw and processed sample data. These folders follow the sample throughout the sample's life at NEA. Ultimately the folders, raw data and computer files are stored and maintained for a minimum of seven years unless otherwise requested by the client. Northeast Analytical's Laboratory Information Management System LIMs also records sample progress throughout the sample life-cycle.

In cases where a legal/evidentiary record of sample custody transfers is required or at the request of the client an Internal Chain of Custody tracking form indicating internal custody transfers is included in the "Sample LRF Folder". An example of the Internal Chain of Custody Tracking form is included in appendix F.

5.0 GENERAL LABORATORY PROCEDURES

5.1 INTRODUCTION

NEA goes to great efforts to produce high quality data by employing the most qualified staff, methods, facility and instruments.

5.2 LABORATORY PHYSICAL SERVICES

Laboratory physical services and instrumentation are maintained in accordance with OSHA, NYS ELAP/NELAP and US-EPA requirements. Specific information regarding these topics can be found in the Laboratory Chemical Hygiene Plan (CHP) and individual instrument or method SOPs.

Laboratory Hoods: Fume Hoods are decontaminated after each use by laboratory staff. They are also cleaned periodically by technicians and support staff. Hood face velocities are measured at intervals prescribed in the CHP.

Laboratory Ultra Pure Water System: NEA utilizes a Continental model Modulab® LB PUO 1002 reverse osmosis (RO) system to supply laboratory type II water. The RO water system is monitored for volume used at each use through a RO system logbook. The specific conductance is also monitored at regular intervals using an independent meter.

5.3 GLASSWARE

All laboratory glassware is thoroughly cleaned and carefully maintained utilizing the most appropriate procedures for the given type of glassware. The cleanliness of glassware and equipment is monitored though the analysis of Method Blanks. Method blanks are routinely run with each analytical/extraction batch. The cleaning procedures are detailed in NEA Standard Operating Procedures (SOPs).

5.4 REAGENTS, SOLVENTS AND GASES

NEA utilizes the highest grades of reagents, solvents and gases for laboratory analysis. Reagents and solvents are initially evaluated and then routinely monitored for target analyte contamination using method and reagent blanks. Gases used by the laboratory are purified for gas chromatography use by passing through absorbent/filtration traps where applicable.

5.5 INSTRUMENTATION

NEA uses the latest state-of-the-art instrumentation. A listing of major NEA instrumentation can be found in Appendix G. All instruments are maintained internally by staff using the routine preventive maintenance schedules outlined in the respective method SOP. Major instruments (ie: GC, GC/MS, ICP, GFAA, etc.) are also maintained through vendor service contracts and parts contracts. General Instrumentation procedures and record keeping is outlined below, further information can be found in method SOPs and Instrument Run and Maintenance Logs.

5.5.1 INSTRUMENT LOG BOOKS

Instrument use as well as routine and non-routine maintenance for major instrumentation is monitored and documented by using Instrument Run Logs and Instrument Maintenance Logs. The Instrument Run Logs document the routine analytical procedures conducted for all samples. All parameters that may impact a sample analysis are recorded to assist in documenting proper analytical procedures and to assist in any troubleshooting. An example of an instrument Run Log is included in Appendix H.

The items addressed in the Logs are outlined below:

- **QA/QC frequency**
  - Calibration
  - Calibration Verification

- Analytical sequence
- Date of analysis
- Instrument operator
- Standards used (using standard codes)

- **Sample analysis**
  - Sample analytical sequence
  - Comments regarding analysis of individual samples
  - Re-analysis, re-extraction information
  - Analytical dilutions (sample volume)

- **Routine maintenance, system performance**
  - Routine maintenance
  - System performance monitoring (ie: purge flow)

The Instrument Maintenance Log also contains information about non-routine maintenance. It is divided into three sections:

1. Description of the Problem
2. Procedures Conducted to Correct Problem / Troubleshooting
3. Problem Resolution / Outcome

This has been found to be a great aid in documenting analytical problems as well as troubleshooting recurring problems. An example of an Instrument Maintenance Log is included in Appendix I.

5.5.2 INSTRUMENT PREVENTATIVE MAINTENANCE

Routine preventative maintenance (PM) is paramount for proper instrument operation and the production of quality data. PM schedules are generated based on manufacturer's recommendations, regulatory requirements and Laboratory SOPs. Preventive maintenance frequency is based on system performance, number of samples analyzed and fixed time periods. Detail for individual PM schedules can be found in method SOPs, and instrument maintenance logbooks.

5.5.3 INSTRUMENT MAINTENANCE AND SERVICE

NEA strives to utilize screening techniques whenever practical to minimize damage to instrumentation due to sample overload or contamination. However, given the unknown nature and matrices of environmental samples analyzed on laboratory instrumentation, troubleshooting and non-routine instrument maintenance will unfortunately be required even under the best conditions. All instrument problems, troubleshooting and maintenance are recorded in the Instrument Maintenance Logbook. Instrument maintenance is performed in-house when possible. If a problem requires further service, a service technician from the vendor is contacted to repair the instrument. NEA maintains vendor service contracts or parts contracts on all major instruments (ie: GC, GC/MS, ICP, GFAA, etc).

6.0 Calibration Procedures / Standards / Frequency

6.1 INTRODUCTION

NEA believes the accuracy of analytical results relies on the use of quality reagents and standards. Reagents and standards are tested for quality and lack of contamination BEFORE use. Outlined in the following sections are the general practices utilized to ensure that standards and reagents are of sufficient quality for analytical use. More detail on these quality assurance practices can be found in the Standard Preparation, individual method and General Laboratory Practices SOPs.

6.2 REAGENTS AND STANDARDS

All chemicals received at the laboratory are labeled with the date received and when containers are opened they are labeled with date opened. Standards are logged into the lab by recording the date received, vendor, vendor code, concentration and standard code in the Standard Receipt Logbook. Reagents and standards are tested for accuracy and lack of target compound contamination when they are received and after any subsequent preparation steps. Reagents and standards are certified by lot. Standard and reagent preparation is recorded in the Standard Prep Logbook along with an expiration date and standard preparation code.

The standard preparation sequence is as follows:

1. Check standard expiration date

2. Fill out Standard Prep Logbook:

a. Date

b. Analyst

c. Standard vendor, standard name, vendor code, vendor lot number, standard expiration date

d. Solvent type, vendor and lot

e. All standard preparation steps

f. Standard code:

MMDDYYBNPnnnA

Where: MM = month
DD = day
YY = year
N = prep. logbook number
nnn = standard prep logbook page
A = letter for standard preparation on the page (ie: first prep = A, second prep = B ...)

g. Prepare a standard label including:

standard contents
standard concentration
standard code
standard solvent
expiration date

3. Prepare standard as outlined in the logbook.

a. Use only class "A" or equivalent volumetric glassware

b. Prepare standard utilizing proper safety equipment (ie: Fume Hood, gloves, etc)

4. Mark the standard "DO NOT USE UNTIL TESTED - analyst initials"

5. Test the standard against a laboratory reference standard. If the standard does not have any target contamination and is within the laboratory specified accuracy criteria do the following:

a. remove the "DO NOT USE ..." label

b. Mark the standard prep logbook with the date tested, analytical queue or batch name and analyst initials.

c. File the analytical raw date and % difference calculations as appropriate.

6.3 STANDARD REFERENCE MATERIALS

To ensure the accuracy of the analytical standards and results, standards prepared in the laboratory are tested against an independent mixture of known quality and accuracy. Standards are first checked by vendor lot for accuracy and lack of contamination BEFORE use. Independent standards may be from one of the following certification programs:

- NIST
- NIST Traceable Vendor
- Other appropriate Certified Source

All subsequent preparation of standards may also be checked against an independent mixture BEFORE use.

6.4. INSTRUMENT CALIBRATION

All instruments and equipment are calibrated and the calibration verified at intervals no greater than specified in the following documents:

- ELAP laboratory manual
- NEA SOP
- Regulatory method
- Vendor recommended calibration interval

All instruments are calibrated BEFORE analytical procedures are run. The calibration range is determined by the highest calibration standard, the lowest calibration standard, and as appropriate, the laboratory determined method detection limit (MDL). In general, all calibrations are verified by running a continuing calibration check standard (CCC) containing all target compounds at the beginning and (if appropriate) the end of each analytical sequence. A CCC standard is also run after every ten samples throughout the sequence unless specified otherwise in the test method. An analytical sequence is deemed to start at the time of the first injection (CCC standard or tuning standard) and continue uninterrupted with repeated analyses until the final CCC is injected or the system remains inactive for greater than 12 or 24 hours as per the analytical method, whichever occurs first. The frequency, nature, tuning, calibration and CCC criteria are outlined in individual test method SOPs.

Laboratory calibration frequency for the following laboratory equipment is outlined below: (for further information, consult the individual equipment SOPs).

	Equipment	**Calibration Frequency**	**Other**
•	Analytical Balances	Daily verification	Annual maintenance from vendor
•	Analytical Balance Calibration Weights	Annually against NBS certified weight by a certified vendor.	
•	pH meters	Daily or with each use	
•	Specific Conductance	Daily or with each use	Cell constant of meters determined annually
•	Thermometers	Verified annually against NBS/NIST traceable certified thermometer	

•	Refrigerators	Temperature verified twice each business day as per NYS ELAP/NELAC requirements
•	Automatic Pipetters	Gravimetrically verified quarterly
•	Glass Microliter Syringes	Gravimetrically verified upon receipt if purchased without certification of accuracy/precision or as necessary based upon inspection for wear/malfunction.
•	Volumetric Glassware	Gravimetrically verified as necessary based upon inspection for wear/malfunction.
•	Other volumetric equip.	Upon arrival or as necessary

6.5 TRACEABILITY OF MEASUREMENTS

A traceability record of all test results to NIST traceable primary reference standards or other applicable certified reference material is maintained with the use of standard preparation logbooks, instrument injection logs, and vendor supplied Reference Standard Certificates of Analysis. Preparation of Calibration Standards, Secondary Dilution Standards, and Spiking Mixtures are documented in standard preparation logbooks with reference to the original vendor lot code. Additions of spiking mixtures to unknown samples and QC samples are recorded in instrument injection logs and/or sample extraction/preparation logbooks.

All laboratory balances are checked for calibration with NIST traceable class 1 weights. All laboratory thermometers used for test methods are calibrated against a NIST traceable certified thermometer.

Traceability of individual analysts and sample preparation technicians to test results is recorded by the initials of the responsible person in the appropriate sample preparation log or instrument injection logbook.

7.0 ANALYTICAL PROCEDURES

7.1 INTRODUCTION

Northeast Analytical, Inc as a member of the NYS ELAP/NELAP program is required to use only ELAP/NELAP approved methods when available for regulatory purposes unless specifically requested otherwise by the client. Most analytical procedures are conducted utilizing the following protocols:

- **US Environmental Protection Agency**
  - National Primary Drinking Water Standards. 40 CFR Part 141; July 1, 1988.
  - EPA 100-400 series - Methods for Chemical Analysis of Water and Wastes: EPA/600/4-79-020, March 1983, EPA-600/R-93/100, August 1993
  - EPA 200 series - Methods for the Determination of Metals in Environmental Samples: EPA/600/4-91-010, June 1991, EPA/600/R-94/111, May 1994

- EPA 500 series methods - Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, December 1988, EPA/600/4-90/020, 1990, EPA/600/R-92/129, August 1992

- EPA 600 series methods - 40 CFR Part 136, July 1, 1988

- Test Methods for the Evaluation of Solid Waste Physical/Chemical Methods (SW-846), Third Edition, Final Update III, December 1996.

- **American Health Association, American Water Works Association, Water Pollution Control Federation**

  - Standard Methods for the Examination of Water and Waste Water. 17th Edition 1989, 18th Edition 1992, 19th Edition 1995

Refer to Appendix J for a complete listing of analytical methods and method reference numbers. Analytical method data quality objectives and criteria are applied as specified in the individual SOPs and regulatory methods.

8.0 DATA COLLECTION, PROCESSING AND ARCHIVING

8.1 DATA COLLECTION

Northeast Analytical, Inc. strives to improve data quality and laboratory efficiency through continued automation. Data from major computer controlled laboratory instrumentation is collected either directly to a Local Area Network (LAN) File Server or initially to the interfaced computer workstation and then transferred to the LAN Fileserver. The data is archived via tape backup automatically each day at the fileserver.

The following automated data collection programs are utilized:

- GC - Waters EMPOWER Chromatography Workstations
- GC/MS - Varian Saturn II Data Acquisition/Processing Software/Target Thruput Processing Software
- CVAA - Leeman chemistry workstation software
- GFAA - Varian SPECTRAA software
- ICP - Smart Analyzer Ciros CDD Workstation software

All manual procedures are documented in laboratory notebooks along with the audit trail information (analyst initials, date performed, etc) and any other relevant information. An audit trail of raw laboratory data is maintained by archiving the raw data and any modifications to it. Any modifications to hardcopy raw data require the analyst to initial and date the modification while keeping the original entry legible.

8.2 DATA PROCESSING

All data is processed in accordance with Laboratory Standard Operating Procedures. Data processing procedures are verified through checks by the analyst, supervisor and laboratory director. Computer calculations are initially validated and then are routinely verified by hand calculation. Processed results are transferred to the Laboratory Information Management (LIMS) system either by automated data capture of instrument data files or by direct manual entry of results. Modifications to results stored in the LIMS system recorded in the audit trail function of the LIMs.

8.2.1 NUMERICAL MANIPULATIONS, MANUAL INTEGRATIONS, AND CALCULATIONS

Procedures for routine numerical calculation and manipulations are outlined in the Standard Operating Procedures for each Methodology. Manual Integrations of acquired sample chromatograms are performed on an as-need basis. Manual integrations for GC/MS analysis are flagged by the chromatography workstation processing software (Target Thruput).

8.2.2 STATISTICAL TERMS AND CALCULATIONS

Outlined below are some of the calculations utilized to process routine statistical parameters:

$$\text{Relative Percent Difference (RPD)} = \frac{[D1 - D2]}{\frac{(D1 + D2)}{2}} \times 100$$

where: D1 = First (Original) Sample concentration
D2 = Second (duplicate) Sample concentration

$$\%\ \text{RECOVERY} = \frac{(SSR - SR)}{SA} \times 100$$

where: SR = Original Sample concentration

SSR = Spiked Sample concentration
SA = Theoretical Spike concentration

8.3 DATA ARCHIVING

All data at NEA is archived for a minimum of seven years unless otherwise requested by the client. Hardcopy data is kept in active files for a period of one year and then is transferred to archive storage. All computer data including raw data and processed data is archived utilizing the Storage Manager® (SSM) software by Seagate/Bartas for the Novell® Local Area Network and by direct disk copy for Millenium/Empower Chromatography Server records. The SSM data archiving software utilizes the advanced algorithms to maintain data archives. SSM writes and manages three complete backup copies of all network files to three individual backup tapes. The computer backup tapes are managed such that at least one copy of all files are maintained off site in a separate archive location to preserve data integrity in the event of a laboratory disaster. Additional information regarding laboratory data archiving can be found in the Network Archive SOP.

9.0 DATA REVIEW AND REPORTING

9.1 DATA REVIEW

Data review quality and data control are important steps in the production of accurate, high quality data. Data Review at the laboratory is a multi-step process where data is thoroughly reviewed by the analyst, section supervisor, and/or QA officer and/or Laboratory Director. If there are any QA/QC or method deviations encountered during analysis or discovered during data review, they are noted and included in the "Sample Progress Folder". Ultimately, all data is reviewed and signed off by the laboratory directory or appointed assistant.

Data Review is routinely conducted as follows:

- **Primary Analyst**

    - Analysis vs. COC specified method
    - Sample history checked
    - Sample analyzed within extraction and analytical hold times
    - Method blank contamination noted
    - All routine deviations annotated
    - Transcriptions checked
    - Reporting units correct
    - Concentrations verified by analyst review
    - Surrogate recoveries within criteria or noted
    - Matrix spikes and duplicates within criteria or noted
    - Instrument tuned within specified limits
    - Calibration curve within criteria
    - Calibration standard has been verified by an independent standard
    - Initial Calibration curve and Check Standards within criteria
    - Analytical sequence within criteria: (standard and QC frequency within criteria)
    - Significant figures checked
    - Sample volumes, conversion factors, and dilution factors checked
    - Solid sample dry weights checked
    - PQLs checked
    - Other Comments

- **Section Supervisor and/or QA officer and/or Lab Director**:

    - Analysis vs. COC specified method
    - Sample history checked
    - Sample analyzed within extraction and analytical hold times
    - Method blank contamination noted
    - All routine deviations annotated
    - Transcriptions checked
    - Reporting units correct
    - Concentration results verified by analyst review
    - Surrogate recoveries within criteria or noted
    - Matrix spikes and duplicates within criteria or noted
    - Instrument tuned within specified limits
    - Calibration curve within criteria
    - Has the calibration standard been verified by an independent standard
    - Initial Calibration and Check Standards within criteria
    - Analytical sequence within criteria: (standard and QC frequency within criteria)
    - Significant figures checked
    - Sample volumes, conversion factors, and dilution factors checked
    - Solid sample dry weights verified
    - PQLs checked
    - Client identification correct on certificate of analysis
    - Client P.O. or billing identification is correct
    - Sample results are reasonable for site history and other factors

- Other Comments

The following areas are also reviewed by the Quality Assurance Officer on a spot check basis.

- **Data Audit by QA Officer:**

  - Chain-of-Custody (COC)

    The COC is used to document all sample transfers, storage and requested analytical methods requested.

    Chain-of-Custody is verified for:

    - Date Sampled
    - Dates of custody
    - Proper signatures of custodians
    - Preservatives
    - Storage location and conditions
    - Sample condition including temperature
    - Analysis vs. COC requested tests

  - Record-Keeping Requirements

    This step of the review process checks to see that the laboratory specified documentation has been completed and documentation procedures have been followed.

    The documentation is verified for:

    - Completeness (all documents filled out and complete)
    - Initialing, dating and annotating of corrections or errors
    - Identification of each piece of documentation with the sample number or study number
    - Identification of chromatograms, graphs, graphical reports with sample numbers
    - Identification of instruments and analysts used during sample analysis

  - Traceability Audit

    The traceability step insures that there is a clear and complete trail of any reported value back to the raw data. This step is often completed as part of the Calculation Audit step that follows.

    The reported values are spot checked against the raw data by checking all calculations from raw data through final result. This ensures that all necessary data is present and the calculations and results are accurate.

    The following items are verified by the document traceability audit;

    - Transcription errors
    - Volumes, conversion, dilution and solid weight factors checked
    - Documentation of all method deviations in the sample narrative, CAR or through QA footnotes or notations

  - Calculations Audit

    The calculations audit provides a method to spot check the accuracy of algebra and arithmetic utilized to calculate the final results. This step is most easily performed

as part of the Traceability step. Five percent of the calculations are spot checked to ensure accuracy.

As part of the calculations audit the following items are examined:

- Reporting units are verified
- Reasonableness of the results are checked
- Significant figures checked
- Reporting limits checked (MDLs or PQLs)

- Analytical Methods and Reference Standards Audit

The use of validated methods and accurate calibration standards is paramount to data quality. Only validated, regulatory approved analytical methods are used unless specifically requested by the client. All documentation is kept available on site to verify how individual steps in the method were carried out, including sample preparation, extraction, cleanup, dilution and analysis. Any deviations from the method specified in the SOP are documented, dated, initialed, and fully explained on a CAR report or comments with raw data results.

- The following method and standard references are verified:

  - Reference to analytical method and standard codes used
  - Discussion of any problems, deviations or changes included on a CAR report
  - Description of the instrument, analytical conditions and analytical procedures
  - Tuning, calibration and calibration check standard procedures and results
    - calibration curve or check standard within laboratory criteria
    - calibration, standard and sample injection sequence outlined and within criteria
  - properly labeled raw data output for all samples and quality control samples
  - quality control results, including: blanks, spikes, duplicate data
    - calibration curve or check standard within laboratory criteria
    - concentration results verified by analyst review
    - compound concentration within calibration curve
    - method blanks within criteria
    - spiked compound recoveries within criteria
    - surrogates recoveries within criteria
  - Any other relevant materials

- Field Data

When available from the client, field data is included in the sample narrative for completeness.

9.2 DATA REPORTING

Results will only be released to the contracting client unless written approval is given by the client. Results will only be reported as preliminary until it is fully reviewed and validated by all levels of review. NEA will report data through hard-copy certificates of analysis, FAX, computer diskettes, e-mail or modem upon client request. Some of the alternate means of reporting have an associated surcharge; please consult the laboratory for additional details. All raw and final results are maintained in the active files for one year and then archived for a minimum of seven years unless specified otherwise by the client. Certificates of analysis are generated which include but may not be limited to the following:

Date Certificate Generated
NYS-ELAP Lab ID#
Sample Collection Date
Sample Receipt Date
Test Method Used
Sample Matrix
Client Sample Identification
Sample Date Analyzed
Concentration results with appropriate units
Detection Limits
Total Number of Pages of Report

9.3 UNCERTAINTY IN MEASUREMENT

Estimation of Uncertainty in Measurement:

Certain Methodologies require an estimation of Uncertainty in Measurement be reported with the analysis data set. For these methodologies the measurement may be reported with a +/- error estimate (error bars). For example a radioactivity measurement might be expressed as 100 microCuries +/- 10 microCuries. For any methodology requiring reporting of an error estimate with the analysis result Northeast Analytical follows the instructions for estimation of error provided in the methodology. For all other methodologies that do not have an estimation of error procedure provided explicitly in the method Northeast Analytical will use the repeat analysis of known standards to determine this information when requested by a client or regulatory body. At least 4 replicates of a known concentration near the midpoint of the calibration range will be analyzed and the standard deviation will be determined. The error estimation of precision for the methodology will be +/-1 standard deviation of the replicates. The Initial and/or Continuing Demonstration of Precision (IDOP) study may be used for this purpose. IDOP data may be pooled to provide a larger statistical population. If error estimates near the method detection limit are requested, concentration measurements of the 8 replicates used to generate the Method Detection Limit may be used for this uncertainty measurement. Again the error estimate of precision will be +/- 1 standard deviation of the replicates. For bias estimation the average percent recovery of the replicate measurements of analyte will be used to estimate bias. For example if the average percent recovery is 85 % then the bias estimate is -15 %. Bias near the midpoint of the calibration and bias near the Method Detection Limit can be determined in the same fashion as the precision estimate using the IDOP and MDL study recovery information.

10.0 LABORATORY QUALITY CONTROL FOR ANALYTICAL PERFORMANCE

10.1 INTRODUCTION

The Quality Assurance program at Northeast Analytical, Inc. was developed to meet or exceed the Quality Control requirements of the NYS- ELAP and EPA programs. NEA seeks to foster a laboratory environment that will encourage all staff to produce high quality results. Through the combined efforts of management, staff and the Quality Assurance Unit (QAU), data quality is continuously planned, monitored, evaluated and corrected when necessary.

The cornerstones of the QA program are:

- Documentation (ie: QAP, SOPs, training logs ...)
- Continuous training and supervision
- Two-way communication and evaluation between analysts and management
- Clearly defined QA/QC criteria
- QA/QC and lab protocol compliance monitoring (ie: use of surrogates, spikes, checks and internal audits)
- Initial Demonstration of Precision and Refinements and improvements of method after initial validation.
- Performance Evaluation Samples
- Internal Audits
- Annual Review of Quality Systems and Corrective Actions

The remainder of this document will outline the protocols utilized to evaluate and report NEA laboratory results.

10.2 LABORATORY SAMPLE QUALITY CONTROL

10.2.1 SAMPLE CONTAINERS

NEA supplies sample containers to its clients. Sample containers are purchased pre-cleaned, and may be tested and certified by the sample container vendor. Pre-cleaned containers receive no additional testing for contamination other than the analysis of trip and holding blanks. This is outlined in the sample container receipt, storage and QA- SOP.

10.2.2 LABORATORY TRIP BLANKS

When NEA is requested to sample for volatile organic compounds a laboratory trip blank is prepared and carried though all transportation and sampling procedures. The purpose of the trip blank is to evaluate possible contamination from sample transport through sample analysis. NEA highly recommends that clients utilize trip blanks during their own sampling events and laboratory generated trip blanks are supplied upon client request.

10.2.3 LABORATORY HOLDING BLANKS (LHB)

In the Laboratory holding blank (LHB) program VOA holding blanks are setup monthly and subjected to the same storage and analytical procedures as the client samples. This allows the laboratory to evaluate container, storage and analytical sources of laboratory contamination.

10.2.4 LABORATORY SOLVENT BLANKS (LSB)

Laboratory solvents used for sample extraction and preparation are may be tested before use to prevent the contamination of sample extracts from contaminants contained in extraction/dilution solvents. Solvents are tested by vendor production lot.

10.2.5 LABORATORY METHOD BLANKS (LMB)

Laboratory method blanks are extracted with all analytical extraction batches utilizing

exactly the same reagents, solvents and procedures to assess possible laboratory contamination incurred during the extraction and analysis steps. This tool is used to ascertain if analyte concentrations in the extraction *batch* are due to contamination. An example of the method blank QA worksheet is included in Appendix L.

If the method blank results are above the analyte detection reporting limit corrective action is performed. When necessary, the client is informed and given the option to decide if the associated samples are to be:

- Re-extracted/Re-prepared (if possible)
- Noted for method blank contamination (B –flagged) if re-extraction/re-preparation is not applicable or the client does not request re-extraction
- Re-sampled and re-submitted to the laboratory for analysis.

10.2.6 LABORATORY SAMPLE DUPLICATES

Laboratory sample duplicates are designed to evaluate the analytical precision of the extraction, analyst and instrument for the associated *batch* of samples. Samples are split at the laboratory and receive complete replicate analysis. The results of the duplicate analysis are evaluated for relative percent difference (RPD), and evaluated for conformity to laboratory or method specified criteria. An example of the laboratory sample duplicate QA worksheet is included in Appendix M. Samples are selected for duplicate analysis at random when a-priori knowledge of sample concentrations is not available. If possible, samples known to contain target analytes corresponding to the test method are chosen to provide statistical measurement of duplicate precision.

10.2.7 SAMPLE MATRIX SPIKES (MS) AND MATRIX SPIKE DUPLICATES (MSD)

Sample matrix spikes (MS) and matrix spike duplicates (MSD) are designed to evaluate both the precision and accuracy of the analytical method *batch* as applied to the various sample matrices encountered. For matrix spikes, homogeneous samples are split at the laboratory, one sample is spiked with a solution containing the analytes of interest. Both original sample and spiked sample are analyzed and the matrix spike percent recovery is evaluated for conformity with laboratory or method specified criteria. Matrix spike duplicates (MSDs) are replicate spikes on the same sample. MSDs are evaluated for percent recovery and relative percent difference (RPD) with respect to the MS. An example of a Matrix Spike / Matrix Spike Duplicate worksheet is included in Appendix N. Matrix spikes are performed at the frequencies prescribed by the analytical method and NYS ELAP and NELAP requirements, one spiked sample per every 10 analyses (or one MS/MSD set per 20 samples) unless otherwise specified.

10.2.8 LABORATORY CONTROL SPIKES (LCS) AND LABORATORY CONTROL SPIKE DUPLICATES (LCSD)

Laboratory control spikes (LCS) are performed to provide accuracy data about the analytical method batch. An aliquot of a prepared spike solution is added to a contaminant free matrix. Corrective action including sample re-processing/re-extraction and/or data flagging may be required for Lab Control Spike failures as indicated by lab SOP and Method requirements. Lab Control Spike Duplicates (LCSD) are performed to provide accuracy and precision data about the analytical method *batch* in instances where not enough sample has been provided to perform matrix spike/matrix spikee duplicate analysis on an actual sample. An aliquot of a prepared spike solution is added to a contaminant free matrix. The percent recovery and relative percent difference are calculated for LCS and LCSDs.

10.2.9 SURROGATE SPIKES

NEA spikes all samples for organic analysis with a surrogate compound unless otherwise

specified in the analytical method. Surrogate spikes evaluate the efficiency and accuracy of *individual* extractions and analyses. Surrogate compounds are chosen which mimic the behavior of the target analyte(s) during the analysis. Surrogate compounds may be prescribed by the test methodology and are spiked into each sample at appropriate concentration prior to the extraction/analysis. Surrogate percent recoveries are evaluated for conformity with laboratory criteria or method specified criteria.

10.3 LABORATORY QUALITY CONTROL LIMITS AND CRITERIA

10.3.1 ANALYST/METHOD INITIAL AND CONTINUING DEMONSTRATION OF COMPETENCY (PRECISION AND ACCURACY)

The precision and accuracy (P&A) of a method is established prior to the analysis of any samples. In general, four replicate samples each consisting of a standard spike of known concentration in a contaminant free matrix are analyzed. The results for percent recovery and relative standard deviation are compared to acceptance criteria for the specified method on a per analyte basis. The procedure is performed for each analyst/analytical instrument and each matrix / extraction method combination and is repeated as required by changes in instrumentation or methodology. Initial P&A acceptance criteria must be satisfied before analysis of samples may begin and before the analyst/technician may perform the method without supervision. Continuing demonstration of proficiency is accomplished by the analysis of blind samples or repeat of initial demonstration of competency procedures.

10.3.2 METHOD DETECTION LIMITS (MDL)

The method detection limit (MDL) is defined as the constituent concentration that when processed through the entire method produces a signal with a 99% probability that the true value is greater than zero as defined in the Code of Federal Regulations, 40 CFR, Part 136 Appendix B (7-1-91). The MDL is established by analyzing a minimum of 7 replicate spikes of the target analytes spiked at a level that produces a signal approximately 5 times the level of the background signal. MDLs are established on a per-instrument, per-matrix, per-analytical method basis. MDLs are re-established as changes in instrumentation and methodology warrant. MDLs are verified periodically by measurement of low level reference standards as required by the Methodology or per NELAP.

10.3.3 METHOD PRACTICAL QUANTITATION LIMIT (PQL)

The method PQL is defined as the lowest level of the constituent concentration that can be quantified in actual sample matrices using routine laboratory operating conditions. The PQL is highly matrix dependent and is generally established at approximately 5 times the MDL or as the equivalent of the lowest calibration standard employed by the method. Adjustments to the PQL are made as warranted by the background interferences and any required dilutions of actual samples. PQLs are established as the equivalent concentration of the lowest calibration standard for methodologies that specify this requirement.

10.3.4 METHOD PERFORMANCE LIMITS

Method performance is routinely monitored as specified by EPA methodology and NYS ELAP/NELAP regulations. Statistical derived control limits are established for matrix spikes, surrogate spikes, laboratory control spikes, and reference samples. Control limits are established for each parameter at the onset of a newly established method. The limits are updated annually or as warranted when changes are made to procedures or methodologies.If an analytical result exceeds a control limit, the measurement is repeated. If the repeat analysis confirms a problem, a Corrective Action Report (CAR) may be issued by the analyst or supervisor. The CAR lists the samples involved, the cause of the problem, action taken to correct the problem and preventive actions to prevent future occurrences. The analyst, supervisor, and QA officer review, approve and sign the report.

10.4 LABORATORY PERFORMANCE, SYSTEM AUDITS AND CERTIFICATION.

10.4.1 INTERNAL LABORATORY AUDITS

Internal Audits are distinguished from Independent Audits in that internal audits are initiated in house and are designed to measure compliance with in house established criteria. Internal Audits may consist of Performance Audits, System Audits, and other devices to measure data quality.

10.4.1.1 SYSTEM AUDITS

System audits are performed at scheduled intervals and as warranted by encountered circumstances. System audits may consist of qualitative evaluations of all quality control measurement systems or of selected sub-units. System audits consist of written evaluations of quality control documentation and record keeping, spot checks of procedural compliance with Standard Operating Procedures, review of data generated by quality control samples, review of test documentation and review for compliance with laboratory ethics requirements. System audits are designed to determine whether activities scheduled by the QA Unit are satisfactorily performed. A record of any corrective action taken in response to the audit is maintained. See Appendix O for an example of a System Audit Evaluation Report (SAER). Further detail about laboratory audits can be found in the Laboratory QA SOP.

10.4.1.2 PERFORMANCE AUDITS

Performance Audits are conducted at the discretion of management and/or the QA Unit. A performance audit is a quantitative evaluation of the measurement systems for an analytical procedure. Performance audits consist of the submission of samples of known composition under the auspices of the QA Unit with or without the knowledge of the analyst. Single-blind and double-blind performance audits also document whether the analytical method is performed in accordance with the standard operating procedures applicable to the method. The results of the analysis are compared with the known values for conformity with laboratory established acceptance criteria. See the Laboratory QA SOP and Appendix P for an example of Performance Audit Report (PAR).

10.4.1.3 AUDIT CORRECTIVE ACTIONS FOLLOW UP

Corrective actions resulting from internal audits will be discharged in a agreed time frame which at the discretion of management and the QA Unit may be as much as one year. Follow up of corrective actions will be maintained with original audit documentation.

10.4.1.4 RESULTS OF AUDIT DISCLOSURE TO CLIENTS

In cases where finding of an internal audit procedure result in corrective actions that effect data that has already been reported to clients, the client is notified immediately in writing and revised data is generated wherever warranted.

10.4.2 INDEPENDENT AUDITS

NEA is routinely audited by regulatory agencies (i.e.: NYS ELAP), laboratory clients, and agents contracted by laboratory clients. Independent audits are conducted as scheduled by a regulatory agency or at client request. Independent Audits are conducted in accordance with or under the supervision of the auditing agent. A record of the audit and any corrective actions taken in response to the audit is maintained. Results of independent audits are discussed during the annual review of quality systems.

10.4.3 CERTIFICATIONS

NEA is currently certified through the NYS ELAP program for the methods and analytes outlined in Appendix J. In cases where samples may be received from States other than New

York State, notification is made to prospective clients as to the current status of certification. All certificates are displayed and new or revised certificates are made available as soon as they become active. NEA maintains certification in other states on an as need basis for client projects. Reciprocal certification between the NYS-ELAP program and several other state certification programs exists.

10.4.3.1 NYS-ELAP AND NELAP CERTIFICATION

NYS ELAP/NELAP certification is maintained for all applicable analytes. NYS ELAP/NELAP certification requires an biannual on-site inspection of the laboratory facilities and satisfactory analysis of Performance Evaluation (PE) samples. PE samples are submitted by ELAP on a biannual schedule. Copies of NEA's ELAP/NELAP certificates are contained in Appendix Q.

10.4.4 PERFORMANCE EVALUATION SAMPLES

Northeast Analytical participates in the NYS-ELAP/NELAP performance evaluation sample program where samples for submitted biannually for testing. The ELAP PE samples test both Potable Water and Non-Potable Water categories along with Solid and Hazardous Waste categories twice annually.

Additional proficiency samples associated with client specific approval programs are also analyzed as specified in client specific agreements.

10.5 LABORATORY CORRECTIVE ACTION

10.5.1 CORRECTIVE ACTION REPORTING AND TRACKING

Laboratory corrective action is warranted whenever there is a major or systematic excursion from standard operating procedures or when laboratory Quality Control criteria have been exceeded due to a systematic problem. Examples of circumstances that may warrant corrective action are: quality control samples exceeding limits, sample loss during processing, instrument calibration checks exceeding acceptance criteria, etc. The analyst or the party that detects the non-compliant condition may initiate a corrective action report (CAR) using their professional discretion. The initiator may be the analyst, the QA officer, section supervisor, or laboratory director. The report details a description of the problem, steps taken to investigate the problem, steps taken to remedy the problem, steps taken to prevent a recurrence and a review and signature by the supervisor, analyst, and QA officer. Corrective action reports are tracked numerically and copies may be retained with the client files and the QA files for future reference. See Appendix K for an example of a CAR.

10.5.2 CORRECTIVE ACTION FOLLOW UP

Within a period of approximately 3 months after the issuance and resolution of a corrective action report, the supervisor or QA officer initiates a follow up investigation. The follow up consists of a review of the CAR and a determination of whether the solution has been properly implemented and has been successful. The follow up is documented and is attached to the original CAR report.

10.5.3 ALLOWED EXCURSIONS FROM STANDARD OPERATING PROCEDURES

In cases where excursions from test methods or standard operating procedures are necessary, full documentation of the allowed excursion will be maintained with the project work order and test results in the laboratory archives. In some cases, a test method may be reported as modified. Information about test method modifications and excursions is generally described in a sample case narrative that is included with the project data summary report. All excursions and modifications to test method will be made with full disclosure to the client, data user, and/or regulatory agency.

10.6 LABORATORY ADMINISTRATIVE ACTIONS AND CONTROLS

10.6.1 REVIEW OF REQUESTS TENDERS AND CONTRACTS

An assessment of Laboratory Capabilities is undertaken when new projects are proposed. New projects may include analytical methods and techniques that the laboratory has not previously employed or may constitute projects of fixed or indefinite quantities of samples for test-methods currently in use by the laboratory. The following concerns are addressed before the acceptance and commencement of the project:

10.6.1.1 Adequate facility physical space for sample acceptance, initial processing, and archival storage.

10.6.1.2 Adequate qualified personnel to perform the given test methods and meet project specified schedule and data quality objectives.

10.6.1.3 Adequate analytical instrumentation and backup instrumentation to perform the given test methods and to meet project specified schedule and data quality objectives including required detection limits.

10.6.1.4 Sufficient sources of vendor supplied laboratory consumables for the duration of the project.

10.6.1.5 Appropriate Laboratory Certification held or proposed interim certification to be acquired

10.6.1.6 Appropriate laboratory documentation in place including Demonstrations of Proficiency, Method Detection Limit Studies, and Standard Operating Procedures.

The assessment may be conducted formally or informally with senior management and department technical supervisors during regularly scheduled management meetings and/or specially scheduled department meetings.

10.6.2 Laboratory Annual Review of Quality Systems

The Laboratory Quality Systems are reviewed annually this review consists of meeting(s) with Senior Management, QA Personnel, and Section Supervisors. The annual review document encompasses the following items:

10.6.2.1Outcome of Internal Audits

10.6.2.2 Results of assessments by external bodies

10.6.2.3 Results of inter-laboratory comparisons and proficiency tests

10.6.2.4 Changes in workload volume and laboratory capacity

10.6.2.5 Feedback from clients

10.6.2.6 Corrective actions

10.6.2.7 Other factors

10.6.3 Client Notification of Results of Internal Audits and Annual Review

In the event that the findings of an internal audit or annual review affect analytical results previously delivered to a client, the client is notified immediately in writing of the outcome of the audit and how specific results may be affected. Corrected results are issued for affected data.

11.0 DOCUMENT CONTROL

The following Document Control Procedures are employed for laboratory-generated items:

11.1.1 SOP Master List

Standard Operating Procedures are indexed with an SOP Master List. The Master List records the SOP name, SOP file ID, the author, the date effective, and the date withdrawn. All prior versions of SOPs are stored in the Laboratory Data Archives. Laboratory SOP copies are signed out to laboratory personnel and laboratory section manuals which are maintained in the appropriate work areas. Outdated SOP copies are returned and destroyed. The original master copy of retired SOPS are archived in long term storage.

11.1.2 Controlled Documents Master List

Controlled Documents are indexed with a Controlled Documents Master List. Controlled documents include the Laboratory Quality Manual, Logooks, miscellaneous forms, and .other documents. Controlled Documents are identified by a revision, revision date, and controlled paper color.

11.1.3 Archival Procedures

Archived Documents such as the Quality Assurance Manual, Retired Standard Operating Procedures, Chemical Hygiene Plan, and outdated Test Method Manuals are stored in the Laboratory Information Archives. A Laboratory Information Archive Master List is maintained for appropriate items.

11.1.4 Document Storage and Access Log

Archived hardcopy materials including raw data, processed data, and laboratory logbooks are stored in a secured onsite data storage area (Laboratory Data Archives). Access to the data storage area is limited to authorized personnel. Items that are removed from the data storage area are signed out in an Access Log and are signed back in when returned.

11.1.5 Hand Amendments to controlled documents

Controlled documents especially Standard Operating Procedures (SOPs) will periodically require hand annotations in order to keep the documents current (updates) and to correct errors discovered in the original document. Any hand amendment to a controlled document requires an initial and date by an authorized signatory. Authorized signatories include department managers/supervisors, the laboratory manager, laboratory director and QA officer.

12.0 DATA INTEGRITY PLAN

Northeast Analytical Inc. is dedicated to the production of the data of the highest quality. The Data Integrity Plan is employed to ensure that each employee is informed of ethical requirements for data production and that fraudulent activities may result in termination of employment and/or possible civil/criminal prosecution. The Data Integrity Plan also provides mechanisms for ensuring that ethical behavior is promoted and is a routine part of laboratory operations:

12.1 Ethics Training

Each employee is required to attend an initial training session provided by Northeast Analytical Inc. regarding proper ethical behavior in the laboratory environment. The need for honesty and full disclosure in all analytical reporting and record-keeping is emphasized. Topics covered during this training session, including handouts, are kept on file in the Quality Assurance Department. Copies of signed training session documentation are kept with employee training records. Refresher training sessions are required for laboratory personnel as new topics are added the seminar or once annually.

12.2 Ethics and Data Integrity Agreement Statement

Each Employee is required to sign the laboratory *Ethics and Data Integrity Agreement* Statement (Please see Appendix for example)

12.3 Data Integrity Advisor

A Data Integrity Advisor is appointed by senior management to direct management of the need for any detailed investigation. The data integrity advisor ensures confidentiality and ensures a receptive environment to privately discuss personnel ethical dilemmas with staff or observed unethical practices by other members of staff. Confidential records of these discussions are kept on file.

12.4 Monitoring

The Quality Assurance Officer audits at least 5 percent of generated laboratory data packages on a quarterly basis to ensure that data integrity requirements are met. Blind reference samples may be employed to test quality systems as deemed necessary by Senior Management and /or the Quality Assurance Officer.

12.5 Documentation

All data integrity incidents are documented with secure electronic files and locked filing cabinets. Documentation includes investigative findings and disciplinary actions. If client disclosure is deemed necessary by senior management then such disclosures and investigations are recorded.

13.0 APPENDICES

A. Laboratory Employee Confidentiality Statement

B. Laboratory Organizational Outline

C. Laboratory Personnel Qualifications

D. Employee Training Log

E. NEA Chain-of-Custody Document

F. NEA Internal Chain of Custody

G. NEA Instrumentation Listing

H. Instrument Run Logbook Example

I. Instrument Maintenance Logbook Example

J. NEA Analytical Method Listing

K. Corrective Action Report (CAR) Example

L. Laboratory Method Blank Form

M. Laboratory Sample Duplicate Form

N. Laboratory Matrix Spike / Matrix Spike Duplicate Form

O. System Audit Evaluation Report (SAER)

P. Performance Audit Report (PAR)

Q. NYS- ELAP/NELAP Certifications

R. Laboratory Test Method References

APPENDIX A

LABORATORY EMPLOYEE ETHICS AND CONFIDENTIALITY STATEMENTS

NEA Ethics and Data Integrity Agreement

1. I, _____ (print), state that I have read SOP NE224_01 and understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Northeast Analytical (NEA).

2. I agree that in the performance of my duties at NEA:

a. I shall not intentionally report data values that are not the actual values obtained.
b. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses.
c. I shall not intentionally represent another individual's work as my own.

3. I agree to inform NEA management of any accidental reporting of non-authentic data by myself in a timely manner.

4. I agree to inform NEA management of any accidental or intentional reporting of non-authentic data by other employees.

Employee Signature _____ Date: _____

Employee Initials _____

Q:\forms\NEA Ethics and Data Integrity Agreement.doc rev.00, 6/26/06, QC

Confidentiality

Northeast Analytical, Inc. has engaged in confidentiality agreements with numerous clientele and these agreements are inclusive to all visitors and employees of this company.

Federal regulations require that all visitors and employees of this company comply with this confidentiality agreement; failure to do so will result in immediate dismissal and possible Federal infractions.

I, the undersigned, do understand and hereby agree to comply with the above statements, and I will not divulge any information pertaining to this company's business and activities to anyone or any organization which is not directly associated with Northeast Analytical, Inc.

EMPLOYEE / VISITOR: _____ DATE: _____

TITLE: _____

NEA REPRESENTATIVE: _____ DATE: _____

TITLE: _____

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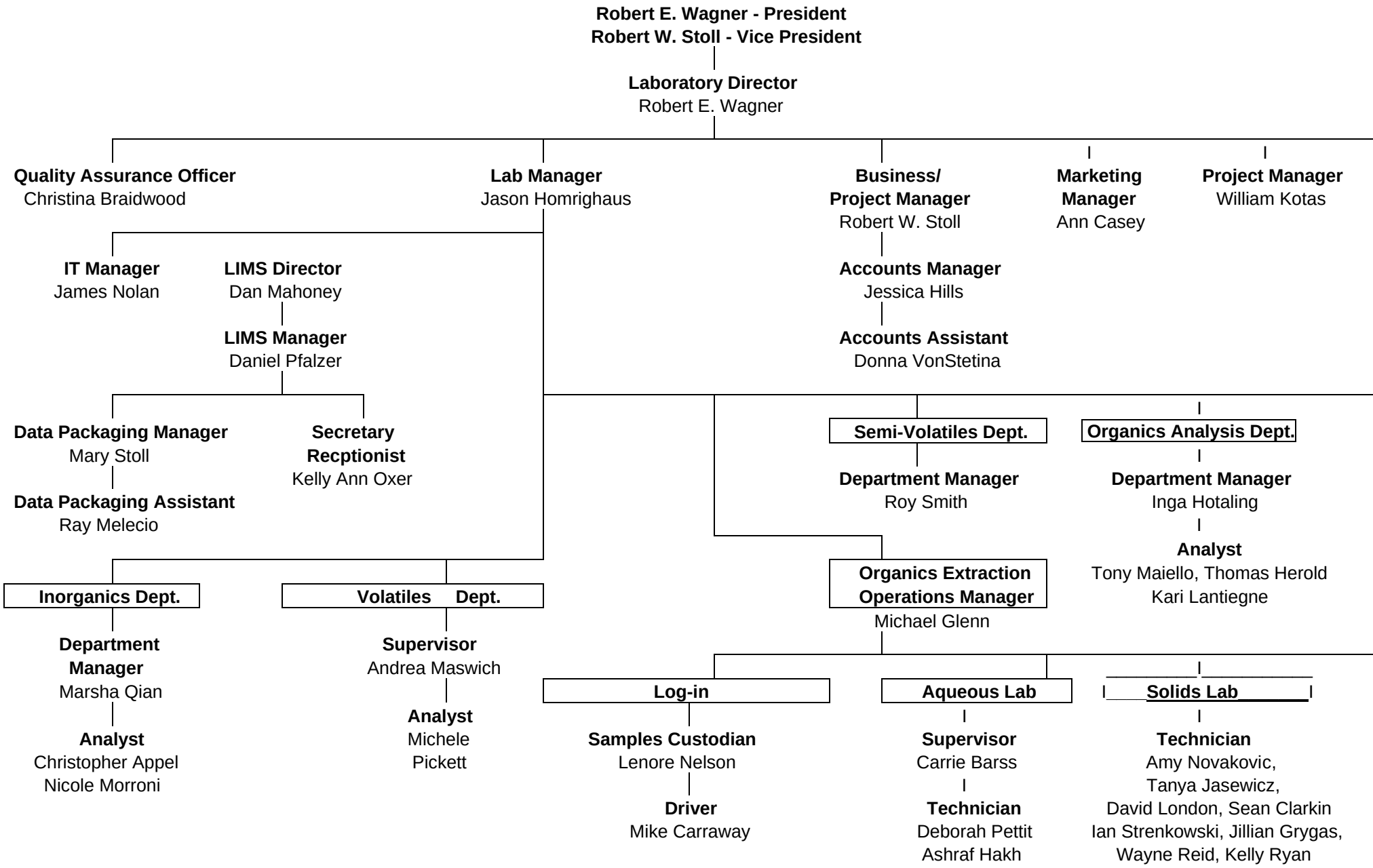
APPENDIX B

LABORATORY ORGANIZATIONAL OUTLINE


Robert E. Wagner - President
Robert W. Stoll - Vice President
Laboratory Director
Robert E. Wagner
Quality Assurance Officer
Christina Braidwood
Lab Manager
Jason Homrighaus
Business/
Project Manager
Robert W. Stoll
Marketing
Manager
Ann Casey
Project Manager
William Kotas
IT Manager
James Nolan
LIMS Director
Dan Mahoney
LIMS Manager
Daniel Pfalzer
Accounts Manager
Jessica Hills
Accounts Assistant
Donna VonStetina
Data Packaging Manager
Mary Stoll
Data Packaging Assistant
Ray Melecio
Secretary
Recptionist
Kelly Ann Oxer
Semi-Volatiles Dept.
Department Manager
Roy Smith
Organics Analysis Dept.
Department Manager
Inga Hotaling
Analyst
Tony Maiello, Thomas Herold
Kari Lantiegne
Inorganics Dept.
Volatiles Dept.
Organics Extraction
Operations Manager
Michael Glenn
Department
Manager
Marsha Qian
Analyst
Christopher Appel
Nicole Morroni
Supervisor
Andrea Maswich
Analyst
Michele
Pickett
Log-in
Aqueous Lab
Solids Lab
Samples Custodian
Lenore Nelson
Driver
Mike Carraway
Supervisor
Carrie Barss
Technician
Deborah Pettit
Ashraf Hakh
Technician
Amy Novakovic,
Tanya Jasewicz,
David London, Sean Clarkin
Ian Strenkowski, Jillian Grygas,
Wayne Reid, Kelly Ryan

APPENDIX C

LABORATORY PERSONNEL QUALIFICATIONS

APPENDIX C: NORTHEAST ANALYTICAL SENIOR MANAGEMENT

Critical to the success of any organization is the caliber of its people. Our laboratory is staffed with highly experienced and committed environmental professionals. NEA employs a multi-disciplinary staff of experienced chemists, analysts, environmental scientists, project managers and support staff. All chemists and analysts maintain excellent scores on proficiency tests to demonstrate capabilities in instrumentation and methodology. NEA is one of a handful of laboratories to pioneer Congener Specific PCB methodologies. Our staff has 40 years of combined experience in the field of PCB chemistry and analysis.

Robert Wagner Laboratory Director

Mr. Wagner has worked on the cutting edge of Congener specific analysis for the past 24 years. He has worked with Dr. John Brown and Dr. George Frame in developing congener specific analysis and PCB congener identification on DB-1 and CP-Sil 18 Columns. As the Lab Director of NEA he supervises and manages laboratory staff in the chemical analysis of various environmental samples for PCB, pesticides/herbicides, inorganic, GC/MS and volatiles; develops and implements analytical methods and procedures.

Robert Stoll Business Manager

Mr. Stoll is the Financial Manager of NEA. He supervises all accounting and business management responsibilities for NEA.

Ann Casey Marketing and Program Development Manager

Ms. Casey has worked on the cutting edge of Congener specific analysis for the past 18 years. She has worked with Dr. Brian Bush and Dr. Chia-Swee Hong in developing congener specific method analysis for PCBs in air, water, soil, sediment, breast milk, blood/serum, urine and tissues. She coordinates the technical projects and client services for PCBs, Pesticides, PAHs, VOCs, Metals and specialty analysis

Jason Homrighaus Laboratory Manager

Mr. Homrighaus has over 10 years of experience in environmental industry. He supervises and manages the laboratory staff and supervisors in the organic and inorganic departments, project management, analysis, data review and lab monitoring for QA/QC compliance.

William Kotas QA/QC Officer/ Project Manager

Mr. Kotas has over 15 years of experience in environmental analysis and quality assurance. Mr. Kotas holds a New York State Department of Environmental Conservation certified data validator for Organic and Inorganic analysis data summary reports. He is responsible for internal laboratory QC audits of analytical methods, software systems and sample preparation techniques.

Christina Braidwood QA/QC Officer (In-training)

Mrs. Braidwood has 7 years of experience in various laboratories involving chemical research and development. Mrs. Braidwood is Six Sigma and DFSS Trained in statistical analysis and problem solving. She is responsible for internal and external laboratory audits, maintaining compliance of analytical methods, and evaluates laboratory quality performance.

Inga Hotaling Congener Specific Specialist

Ms. Hotaling has worked in environmental analysis for the past 14 years. She performs High Resolution PCB congener analysis, data processing and reporting for a variety of matrices. She is responsible for Laboratory Chromatography Database System management and over sees the data generated for the organic section.

Anthony Maiello 8082 & 8081 Specialist

Mr. Maiello has worked in environmental analysis for the past 18 years. He performs the data process and reports all 8081(Pesticides) and 8082 (Aroclor) samples for a variety of matrices.

Mike Glenn Extraction Lab Manager

Mr. Glenn supervises environmental extractions. He supervises and manages the extraction technicians involved in the extraction of soil, sediment, tissue and water samples for PCB, pesticides, herbicides and semi-volatile analysis. He will assign and oversee the technicians extracting the samples.

Marsha (Xia) Qian Inorganic Laboratory Supervisor

Ms. Qian will perform and oversee the analysis, data processing and reporting for the metal 6010 and 7000, TSS, TDS,

pH, and Oil and Grease.

Roy Smith **High Res. PCB Congener and SVOC Supervisor**
Mr. Smith has worked in environmental analysis for the past 15 years. He is responsible for high resolution PCB Congener and semi-volatile analysis of environmental samples of varying matrices, including soil, oil, water and tissue.

Andrea Maswich **GC/GCMS Volatiles Supervisor**
Ms. Maswich has worked in environmental analysis for the past 5 years. She is responsible for Volatiles analysis by GC/MS and manages data processing and reporting for all samples submitted for this analysis method.

Resumes for Northeast Analytical's Senior Staff are presented on the following pages.

ROBERT E. WAGNER: LABORATORY DIRECTOR

PROFESSIONAL EXPERIENCE:

1989-Present: **Lab Director**
Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, New York

Lab Director of Northeast Analytical, Inc.; supervises and manages laboratory staff in the chemical analysis of various environmental samples for PCB, pesticides/herbicides, inorganic, GC/MS and volatiles; develops and implements analytical methods and procedures; instruct laboratory analysts; project manager responsibilities; responsible for review of all data and QA/QC programs; long range planning; client relations; equipment evaluation and recommendation; maintain laboratory certification programs.

1981-1989: **Associate Staff Analytical Chemist**
General Electric Company
Research & Development Center
P.O. Box 8, 1 River Road
Schenectady, New York 12301

Employed in the field of environmental chemistry (PCB, PCDF, DIOXIN) and analytical polymer chemistry (trace analysis, residue solvent characterization, toxic component analysis). Involved with development of multi-component analysis and synthetic chemistry of standards. Isolation and fractionation of environmental residues from complex matrices. Development of state of the art analytical techniques with high resolution capillary gas chromatography utilizing a variety of detectors (ECD, FID, TCD, HECD). Knowledge of GC and HPLC (H/P, Varian, Shimadzu) and integration systems. In-depth maintenance capability to board level and hands-on hardware configuration.

1978-1981: **Biochemistry Project Supervisor**
Albany Medical College of Union University
Albany, New York 12208

Coordinated and developed a federally funded research project investigating the aging process using hamsters as models. Companion study explored the effects of lipid enriched diets on the life span of these animals. Supervised three technicians in the daily work routine. Also conducted clinical research to develop a quick and accurate determination of serum bilirubin levels based on a fluorescence quenching method.

1975-1978: **Biochemistry Technician**
Albany Medical College of Union University
Albany, New York 12208

Technician on above mentioned federal research project. Prepared enzyme complexes from various organ tissue for extensive biochemical analysis. Experience with centrifuges, A.A., Spectrophotometers, and fluorometer.

EDUCATION:

1975: **B.S. Chemistry**
State University of New York, College at Potsdam

PROFESSIONAL AFFILIATIONS:

American Chemical Society
Schenectady Chamber of Commerce

PUBLICATIONS:

1984

J.F. Brown Jr., R.E. Wagner, D.L. Bedard, M.J. Brennan, J.C. Carnahan, R.J. May, and T.J. Tofflemire; "PCB Transformations in Upper Hudson Sediments". Northeast Environmental Science, Vol. 3, No. 384, p.167-179, 1984.

ROBERT E. WAGNER: LABORATORY DIRECTOR

R.E. Wagner and J.C. Carnahan; "Capillary GC-Quantitation of Polychlorinated Biphenyls". Presented before the Varian Chromatography Symposium, April 1984.

1985

B.J. Vivier and R.E. Wagner; "Gas Chromatography Date Reduction Software". GE CR&D Memo Report #MOR-85-024, Class 2, March 1985.

J.F. Brown Jr., R.E. Wagner, D.L. Bedard, M.J. Brennan, J.C. Carnahan, R.J. May, and T.J. Tofflemire; "PCB Dechlorination in Upper Hudson Sediments". Presented before the Division of Environmental Chemistry, American Chemical Society 189th National Meeting, Miami, Florida, Vol. 25, No. 1, Paper #12, p.35-37, April 29-May 3, 1985.

R.E. Wagner, J.C. Carnahan and R.J. May; "Calibration Method for PCB Analysis by Capillary GC and Electron Capture Detection". Presented before the Division of Analytical Chemistry, American Chemical Society 189th National Meeting, Miami, Florida, Paper #201, April 28-May 3, 1985.

R.E. Wagner and J.C. Carnahan; "An Internal Standard for Capillary GC Analysis of PCB Mixtures". Presented before the Division of Analytical Chemistry, American Chemical Society 189th National Meeting, Miami, Florida, Paper #202, April 28-May 3, 1985.

D.L. Bedard, L.H. Bopp, M.J. Brennan, R.E. Brooks, J.F. Brown Jr., J.C. Carnhan, D.T. Gibson, M.L. Haberl, C. Johnson, S.H. Lockwood, K.L. Longley, R.J. May, D.P. Mobley, L.M. Nadium, M.J. Schocker, T. Su, R. Unterman, R.E. Wagner, and G.W. Yeager; General Electric Company Research and Development Program for the Destruction of PCBs, Fourth Progress Report, 6/1/84-6/1/85.

R.E. Wagner and J.C. Carnahan; "An Internal Standard for Capillary GC Analysis of PCB Mixtures". Presented before the General Electric Company-Wide Materials Characterization Symposium, 7th Meeting, April 22-24, 1985.

R.E. Wagner and J.C. Carnahan; "Internal Standard for Capillary GC Analysis of PCB Mixtures". Presented before the Varian Chromatography Symposium, March 1985.

J.F. Brown Jr., D.L. Bedard, L.H. Bopp, J.C. Carnahan, R.W. Lawton, R.D. Unterman, and R.E. Wagner; "Human and Environmental Biodegradation of PCBs". Presented before the EPRI PCB Conference, Seattle, Washington, October 22, 1985.

J.F. Brown Jr., D.L. Bedard, J.C. Carnahan, R.W. Lawton, R.E. Wagner; "Environmental Dechlorination of PCBs". Presented before the Society for Environmental Toxicology and Chemistry Meeting, St. Louis, Missouri, November 11, 1985.

1986

J.F. Brown Jr., R.E. Wagner; "Polychlorinated Biphenyl (PCB) Movement and Transformation in Acushnet Estuary Sediments". A draft manuscript prepared for RTE Aerovox Corporation and GE/CEIP to be eventually written into two or more scientific paper discripting the above title. 9/26/86.

1987

D.L. Bedard, M.J. Brennan, R.E. Wagner, M.L. Haberl, and J.F. Brown Jr.; "Extensive Degradation of Aroclors and Environmentally Transformed PCBs by ALCALIGENES EUTROPHUS H850". Applied and Environmental Microbiology, Vol. 53(5), p.1103-1112, May 1987.

J.F. Brown Jr., R.E. Wagner, H. Feng, D.L. Bedard, M.J. Brennan, J.C. Carnahan, and R.J. May; Environmental Dechlorination of PCBs". Environmental Toxicology and Chemistry, Vol. 6(8), p.579-593, August 1989.

J.F. Brown Jr., D.L. Bedard, M.J. Brennan, J.C. Carnahan, H. Feng, R.E. Wagner; "Polychlorinated Biphenyl Dechlorination in Aquatic Sediments". Science, Vol. 236, p.709-712, May 1987.

J.F. Brown Jr., J.C. Carnahan, S.B. Dorn, J.T. Groves, W.V. Ligon, Jr., R.J. May, R.E. Wagner, and S.B. Hamilton; "The Levels of Toxic PCDF Congeners in Used and Unused PCB Dielectric Fluids". Dioxin '87 - Seventh International Symposium, Las Vegas, Nevada, Oct. 4-9, 1987.

ROBERT E. WAGNER: LABORATORY DIRECTOR

J.F. Brown Jr., and R.E. Wagner; "PCB Dechlorination in Marine Sediments". Abstracts of Papers at the 8th Annual Meeting of the Society for Environmental Toxicology and Chemistry, Pensacola, FL, No. 136, 1987.

1988

J.F. Brown Jr., R.W. Lawton, M.R. Ross, J. Feingold, R.E. Wagner, and S.B. Hamilton; "The Persistence of PCB Congeners in Normal and PCDF-Poisoned Humans". Presented before the DIOXIN '88 Conference, Umea, Sweden, August 21-26, 1988.

J.F. Brown Jr., and R.E. Wagner; "A Simple Index for Summarizing the Alteration State of Environmental PCB Mixtures". Presented before the Society for Environmental Toxicology and Chemistry Meeting, November 1988.

J.F. Brown Jr., J.C. Carnahan, S.B. Dorn, J.T. Groves, M.V. Ligon, R.J. May, R.E. Wagner, and S.B. Hamilton; "Levels of Bioactive PCDF Congeners in PCB Dielectric Fluids from Capacitors and Transformers". Chemosphere.

J.F. Brown Jr., R.E. Wagner, and D.L. Bedard; "PCB Dechlorination in Hudson River Sediment". Science, Vol.240, 1988, p.1674-1676.

1990

J.F. Brown Jr., and R.E. Wagner; "PCB Movement, Dechlorination, and Detoxication in the Acushnet Estuary". Environ. Tox. and Chem., Vol.9, p.1215-1233, 1990.

1996

George M. Frame, R.E. Wagner; James C. Carnahan, John F. Brown Jr., Ralph J. May, Lynn A. Smullen, and Donna L. Bedard "Comprehensive, Quantitative, Congener Specific Analyses of Eight Aroclors and Complete PCB Congener Assignments on DB-1 Capillary GC Columns". Chemosphere, Vol. 33, No. 4, pp. 603-623, 1996.

2005

Casey, A.C., Wagner R.E., Hotaling I.C., and Carlson H. (2005). Application of Automated Solid Phase Extraction for Determination of Polychlorinated Biphenyls (PCB) in Water. Presented before the Annual Conference on Soils, Sediments and Water University of Massachusetts at Amherst

2006

Casey, A.C., Wagner R.E., Hotaling I.C., and Carlson H. (2006). Application of Automated Solid Phase Extraction for Determination of Polychlorinated Biphenyls (PCB) in Water Presented with Horizon Technology at PITTCON 2006

Casey, A.C., Wagner R.E., Herold T. E. and Glenn M. (2006) The Evaluation of Extraction and Cleanup Methods for the Determination of PCB Aroclors in Caulking and Sealing Material. Presented before the Annual Conference on Soils, Sediments and Water University of Massachusetts at Amherst

ROBERT W. STOLL: BUSINESS MANAGER

PROFESSIONAL EXPERIENCE:

1989-Present: **Financial Manager**
Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, New York

Financial Manager of Northeast Analytical, Inc. Supervises administrative personnel; performs all ordering and purchasing; accounting and business management responsibilities; interfaces with daily lab activities; long range planning; client relations; equipment and annual budget regulation; staffing; project manager on laboratory programs.

1985-1989: **Process Engineer**
General Electric Company
Product Operations Department (POD)
Research and Development Center
P.O. Box 8, 1 River Road
Schenectady, New York 12301

Responsible for the operation of the Thin Film Deposition Area of the liquid Crystal Display Pilot Line. Characterization and process control on all films used in the fabrication of Thin Film Field Effect Transistors using Sputtering, Evaporation, and PECVD. Responsible for the daily work routine of four technicians, as well as the equipment, facilities and operator specification for the US factory design.

1980-1985: **Associate Staff Chemist**
General Electric Company
Research and Development Center
P.O. Box 8, 1 River Road
Schenectady, New York 12301

CVD Tungsten Project: Developed a unique CVD reactor that was capable of selectively depositing tungsten in 2 micron, straight side wall vias; at deposition rates in excess of 1000A/min. The technology was sold to GENUS Corp. Developed both selective and non-selective tungsten processes using surface pre-treatments to either enhance or hinder deposition. Analyzed and developed a method of relieving the tungsten film stress which was responsible for the spontaneous delamination of tungsten films on glass surfaces.

Fumed Silica Project: Designed a high temperature infrared flow cell (750C) and a fluidized bed reactor to study the kinetics of fumed silica using a variety of silylating agents. Analyses were conducted using a Nicolet 7199 FT-IR spectrometer and a Micromeritics Digisorb 2500 Surface Area Analyzer. Responsible for the operation and maintenance of both machines.

Translex Project: Synthesis of polycarbonate via the melt polymerization method. The polymers were analyzed for impurities and thermal degradation products utilizing GC, HPLC, and NMR.

ROBERT W. STOLL: BUSINESS MANAGER

1979-1980: **Research Technician**
State University of New York at Albany
1400 Washington Avenue
Albany, New York 12222

Provided technical support for a federally funded research project investigating the aging process using hamsters as models. Companion study explored the effect of lipid enriched diets on the life span of these animals. Prepared enzyme complexes from the heart organ for extensive biochemical analysis. Experience with centrifuges, A.A., spectrophotometers, fluorometers, and oxygraphs.

1976-1977: **Chemical Laboratory Technician**
Corning Glass Works
Sullivan Park Research and Development

Painted Post, New York 14870

Responsible for the precision hydrofluoric acid etching of Vycor glass tubes. Operated two computerized lathes used to place layers of BC13, GeCl4, and PCL5 on the inside of the Vycor tubes, which were later drawn into optical waveguide fibers.

1972-1975: **Inventory Management Specialist**
Military Service
United States Air Force
Rank: Sergeant E-4

Non-commissioned officer in charge of the Base Composite Tool Kit Program. Duties involved meeting with representatives of the base maintenance organizations to determine their tool requirements. Maintaining and updating all records for each composite tool kit. Supervised two airman in their daily routine.

EDUCATION:

1979: **B.S. Chemistry**
State University of New York at Albany
Albany, New York

PUBLICATIONS:
Co-author of one journal article and four published proceedings of meetings.

PATENTS:

Method for Treating Fumed Silica (U.S.Pat.4,554,147)

Enhancing the Selectivity of Tungsten Deposition on Conductor and Semi-conductor Surfaces (U.S.Pat.4,552,783)

Selective Chemical Vapor Deposition Apparatus (U.S.Pat.4,653,428)

Method for Selective Deposition of Tungsten by Chemical Vapor Deposition onto Metal and Semi-conductor Surfaces (U.S.Pat.4,471,928)

ANN C. CASEY: MARKETING MANAGER

PROFESSIONAL EXPERIENCE:

2005-Present **Marketing and Program Development Manager**
Director of Client Services
Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, New York 12308

Coordinates data analysis and laboratory results for client's projects assist in planning, recruitment, client relations, and technical data review. Responsible for marketing, advertising and bid evaluations. Interface with government, academic, industry officials, consultants, lawyers, and directors from other laboratories on a regular basis.

2000-2005 **Sr. Analytical Chemist**
Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, New York 12308

Performs GC-ECD analysis of pesticides, PCBs by EPA 8082 and congener specific methods. Working knowledge of method development for Coplanar PCBs by GC-MS and GC-ECD. Conducts research and development for specialty PCB analysis. Maintenance of Agilent GC. Troubleshooting pesticide and PCB projects. Writing SOPs and implementing newly developed methods.

1994-2000 **Project Coordinator**
Research Foundation of the State University of New York
University At Albany
School of Public Health
Rensselaer, New York

Coordinated the analytical analysis and data delivery system for an NIEHS Superfund Basic Research Project investigating the impacts of hazardous waste (PCB, Dioxins, PAH, Fluoride and Metals). Supervised and managed laboratory staff and graduate students. Coordinated data analysis and data interpretation of PCB laboratory results for principal investigators in toxicology, epidemiology, civil and chemical engineering. Developed research protocols for animal exposure to PCB Aroclors and congeners via dermal, inhalation and ingestion exposure. Worked with communities on Environmental Justice issues and education.

1998-1999 **Director of Environmental and Laboratory Planning**
Saint Regis Mohawk Tribe Environment Division
Rt. 37 Community Building
Hogansburg, New York

Coordinated and developed the Performance Partnership Agreement (PPG/PPA) between the Tribe's Environment Division and the USEPA Region 2. Program projects included Clean Air, Clean Water, Wetlands Protection, Solid waste and Core development. Worked with program project department heads to develop and execute work plans, budgets, long-range goals. Worked with Native American community members and leaders to preserve and protect the communities' environment, natural resources and health.

1993-1994 **Laboratory Manager**
Research Foundation of the State University of New York
University At Albany
The Wadsworth Center
Albany, New York

Coordinated the analytical analysis for an NIEHS Superfund Basic Research Project investigating the impacts of hazardous waste (PCB, Dioxins, PAH, Fluoride and Metals). Supervised and managed laboratory staff for

the PCB laboratory. Method development for state of the art congener specific analysis for sample matrices including breast milk, urine, whole blood, serum, adipose tissue, sperm, sediments, air, water and soils. Wrote and implemented SOP's and quality control and quality assurance programs. Knowledge and use of GC-ECD, GC/FTIR/MS, HPLC (HP and Waters). In-depth and daily maintenance of all laboratory equipment.

1991-1993 **Research Technician II**
Research Foundation of the State University of New York
University At Albany
The Wadsworth Center
Albany, New York

Method development for state of the art congener specific and coplanar analysis. Sample matrices included breast milk, adipose tissue and blood. Experience with GC-ECD and GC-MS.

1990-1991 **Research Laboratory Assistant**
Research Foundation of the State University of New York
University At Albany
The Wadsworth Center
Albany, New York

Method development and extraction of breast milk and urine for congener specific analysis. Experience with GC-ECD.

1989-1990 **Animal Caretaker/Laboratory Technician**
Health Research Inc.
New York State Department of Health
Wadsworth Center for Labs and Research
Albany, New York

Technician for the ATSDR Great Lakes PCB program. Prepared, dosed and sacrificed animals in PCB exposures experiments. Dissection of rats and monkeys. Tissue collection and preparation for PCB analysis.

EDUCATION:

1993: **B.S. Biology**
The College of Saint Rose
Albany, New York

1993: **B.A. Sociology**
The College of Saint Rose
Albany, New York

PROFESSIONAL AFFILIATIONS:

Society of Toxicology and Environmental Chemistry (SETAC)
Association of Occupational and Environmental Clinics (AOEC)
National Environmental Health Association

PUBLICATIONS:

Hong CS, Xiao J, Casey AC, Bush B, Fitzgerald EF, Hwang SA. (**1994**) Mono-ortho- and non-ortho-substituted polychlorinated biphenyls in human milk from Mohawk and control women: effects of maternal factors and previous lactation. Arch Environ Contam Toxicol. 1994 Oct;27(3):431-7.

Kodavanti PR, Ward TR, Derr-Yellin EC, Mundy WR, Casey AC, Bush B, Tilson HA. (**1998**) Congener-specific distribution of polychlorinated biphenyls in brain regions, blood, liver, and fat of adult rats following repeated exposure to Aroclor 1254. Toxicol Appl Pharmacol. Dec;153(2):199-210.

ANN C. CASEY: MARKETING MANAGER

Korytko PJ, Casey AC, Bush B, Quimby FW. (**1999**) Induction of hepatic cytochromes P450 in dogs exposed to a chronic low dose of polychlorinated biphenyls. Toxicol Sci. Jan;47(1):52-61.

Casey AC, Berger DF, Lombardo JP, Hunt A, Quimby F. (**1999**) Aroclor 1242 inhalation and ingestion by Sprague-Dawley rats. J Toxicol Environ Health A. Mar 12;56(5):311-42.

Crofton KM, Kodavanti PR, Derr-Yellin EC, Casey AC, Kehn LS. (**2000**). PCBs, thyroid hormones, and ototoxicity in rats: cross-fostering experiments demonstrate the impact of postnatal lactation exposure. Toxicol Sci. Sep;57(1):131-40.

David F. Berger, John P. Lombardo, Peter M. Jeffers, Anne E. Hunt, Brian Bush, Ann Casey and Fred Quimby (**2001**). Hyperactivity and impulsiveness in rats fed diets supplemented with either Aroclor 1248 or PCB-contaminated St. Lawrence river fish. Behavioral Brain Research, Volume 126, Issues 1-2, 29, Pages 1-11

Quimby FW, Casey AC, and Aquette M. (**2005**) From Dogs to Frogs: How Pets, Laboratory Animals, and Wildlife Aided in Elucidating Harmful Effects Arising from a Hazardous Dumpsite. ILAR J. 2005; 46(4):364-9

PRESENTATIONS

Casey, A.C., Bush, B. (**1994**). The Method Development for Serum Extraction QA/QC. Presented before the Great Lakes Research Program. Madison Wisconsin.

Casey, A.C., Bush, B. (**1995**). PCB Contamination of the St. Lawrence Riverway and the Mohawk Ecosystem. Presented before the 22nd Annual Aquatic Toxicity Workshop. St. Andrews, N.B., Canada.

Casey, A.C., Carpenter, D.O., Bush, B., Tarbell, A. (**1996**). Superfund Basic Research Program. Presented before the Great Lakes Research Consortium. Albany, NY.

Casey AC, (**2004**). Organic Contaminant Analysis for Trace Levels in Seafood and Farmed Raised Fish. Presented before the International Boston Seafood Show.

Casey, A.C., Wagner R.E., Hotaling I.C., and Carlson H. (**2005**). Application of Automated Solid Phase Extraction for Determination of Polychlorinated Biphenyls (PCB) in Water. Presented before the Annual Conference on Soils, Sediments and Water University of Massachusetts at Amherst

Casey, A.C., Wagner R.E., Hotaling I.C., and Carlson H. (**2006**). Application of Automated Solid Phase Extraction for Determination of Polychlorinated Biphenyls (PCB) in Water Presented with Horizon Technology at PITTCON 2006

Casey, A.C., Wagner R.E., Herold T. E. and Glenn M. (**2006**). The Evaluation of Extraction and Cleanup Methods for the Determination of PCB Aroclors in Caulking and Sealing Presented before the Annual Conference on Soils, Sediments and Water University of Massachusetts at Amherst

CHRISTINA L. BRAIDWOOD: QUALITY ASSURANCE/ QUALITY CONTROL OFFICER

PROFESSIONAL EXPERIENCE:

2007- Present: **Quality Assurance Officer**
Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, NY 12305

Responsible for the review of analytical procedures for compliance with US EPA, New York State ELAP and other regulations for environmental analysis. Responsible for scheduling and performing QA operations including internal audits and spot checks. Maintains Quality Assurance/Quality Control records for the laboratory. Responsible for 10% review of analytical reports and data summary packages generated by organics and inorganics sections. Responsible for the maintenance of the Chemical Hygiene Plan and instruction of laboratory personnel on OSHA required safety procedures. Responsible for management and verification of proper laboratory waste disposal.

2005-2007: **Product Development Engineer**
General Electric- Plastics
1 Noryl Avenue
Selkirk, NY 12158

Invented a green flame retardant that enhances the flame resistance (FR) properties of polyphenylene oxide laminates for printed circuit boards. Developed a new synergistic flame retardant system that enhanced FR properties for die attach and laminate applications. Led team with development of a reactive calorimeter assembly for process development in copolymer synthesis. Communicated laminate expertise in poplyphenylene oxide-epoxy chemistries to lead manufacturers in Korea and Japan. Commercialized capped polyphenylene oxide intermediates for electronic applications in US, Europe, and Asia. Trained in Six Sigma statistical analysis for product development.

2002-2004: **Organic and Polymer Chemist**
Schenectady International, Inc.
2150 Balltown Road
Niskayuna, NY 12309

Developed a new UV sensitive adhesive and scaled to Pilot Plant, with patent pending. Conducted formulation and processing experiments in organic and polymer chemistry, specializing to specific customer's needs. Constructed a protocol for water solubility testing of alkylphenols for the EPA. Led team with development of solution to corrosion problem in phenol recycling tower.

2001-2002: **Adjunct Professor for Organic Chemistry**
College of St. Rose
432 Western Ave
Albany, NY 12203

Organized organic chemistry lectures for undergraduate students and supervised laboratory experiments. Prepared chemical reagents and set up laboratory equipment for the each section. Assisted students with the use of the NMR and IR spectroscopy. Developed quizzes and graded final laboratory assignments. Provided help sessions for organic chemistry assignments before and after class.

1999-2000: **Laboratory Technician**
General Electric Corporate Research & Development
1 Research Circle
Niskayuna, NY 12309

Assisted the manufacture and development of ultrasound flex product for electronic devices. Inspected for defects associated with the ultrasound flex design. Manufactured ultrasound flex product by utilizing Ultratec scrubber, photoresist applicator, UV exposure, and lactic acid bath strippers. Inspected semiconductor panels for design anomalies for application in mammography. Assembled a database to track breakage of semiconductor panels during fabrication process. Performed experiments such as taking the particle counts in

various areas of the lab to determine unclean areas. Qualified motor-stage microscopes for incorporation of an automatic inspection process.

1998: **Laboratory Technician**
New York State Department of Health
Wadsworth Center
Empire State Plaza
Albany, NY 12237

Prepared RNA radioactive probes through the use of a gel filtration column. Extracted protein by utilizing mini-gel electrophoresis and observed through Western blotting. Analyzed protein by UV/visible spectrophotometry and high performance liquid chromatography. Extracted photosyntetic protein of Daphnia species to analyze protein properties. Examined well water samples from local areas for Giardia lambia and Cryptospiridium levels.

EDUCATION:

2003: **M.S. Chemistry**
Rensselaer Polytechnic Institute
Troy, NY

1999: **B.S. Biochemistry & Molecular Biology**
Minor: Anthropology
State University of New York at Albany
Albany, NY

PROFESSIONAL AFFILIATIONS:

American Chemical Society (ACS)

PATENTS:
Poly(arylene ether) composition, method, and article (US Pat. 20070082987)

Polymer composition, method, and article (INT Pat: WO/2007/044294)

Flame retardant composition and method (US Pat: 20070080330)

Prepreg-Forming Method, prepregs formed thereby, and printed circuit boards incorporating them (US Docket Num: 217337-1)

Poly(arylene ether) compositions (US Docket Num: 218633-1)

Poly(arylene ether) compositions and articles (US Docket Num: 191492-1)

Method of separating a Poly(arylene ether) composition from a solvent, and Poly(arylene ether) composition prepared thereby (US Docket Num: 223261-1)

Flame Retardant Composition and Method for Capped Poly(arylene ether) (US Docket Num: 189972-1)

Poly(arylene ether) Isolation Process, and Isolated Poly(arylene ether) Prepared Thereby (US Docket Num: 223261-1)

TRAINING:

"Regulatory and Data Evaluation", Annual NY & PA Environmental Lab Conference and Exposition, 2007.

Supported NEA at "The 23rd International Conference on Soils, Sediments, and Water.; Amherst, MA, October 2007.

WILLIAM A. KOTAS: PROJECT MANAGER

PROFESSIONAL EXPERIENCE:

1997-Present: **Quality Assurance Officer/Laboratory Representative**
Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, New York 12308

Responsible for review of analytical reports and data summary packages generated by organics and inorganics sections. Review of analytical procedures for compliance with US EPA, New York State ELAP and other regulations for environmental analysis. Responsible for scheduling and performing QA operations including internal audits and spot checks. Maintains Quality Assurance/Quality Control records for the laboratory. Assists current & prospective clients in selection of test methods, sampling requirements and interpretation of test results. Responsible for maintenance of Chemical Hygiene Plan and instruction of laboratory personnel on OSHA required safety procedures. Responsible for management and verification of proper laboratory waste disposal.

1991-1997: **Quality Assurance Officer/Computer Systems Manager**
Northeast Analytical, Inc.
301 Nott Street
Schenectady, New York 12305

Reviews analytical reports and data summary packages generated by organics and inorganics sections. Reviews analytical procedures for compliance with US EPA, New York State ELAP and other regulations for environmental analysis. Maintains Quality Assurance/Quality Control records for the laboratory. Responsible for installation and maintenance of Laboratory Information Management System (LIMS), Novell Local Area Network (LAN) and all worksations and associated peripherals.

1990-1991: **Microscopist/Analyst**
Entek Environmental & Technical Services
Troy, New York

Performed analysis of air and bulk materials for the presence of asbestos fibers by Phase Contrast and Polarized Light Microscopy. Contributed to the development of a comprehensive training examination for new analysts.

1988-1990: **Environ. Analyst/Quality Control Coordinator**
Professional Service Industries
Pittsburgh Testing Lab Division
Albany, New York

Analyzed air and bulk samples for asbestos. Collected and compiled quality control data generated by each analyst. Assisted laboratory manager in lab operations and client relations.

EDUCATION:

1990: **B.S. Physics**
Minor: Chemistry
State University of New York at Albany
Albany, New York

1985: **A.S. Mathematics/Science**
Hudson Valley Community College
Troy, NY

PROFESSIONAL AFFILIATIONS:

American Society of Physics-Sigma Pi Sigma Physics Honors Society

WILLIAM A. KOTAS: PROJECT MANAGER

PUBLICATIONS:

Contributing editor to *Guide to Environmental Analytical Methods* 4th ed. A summary of Quality Assurance requirements and other pertinent information for select analytical methodologies. Published by Genium Publising Corp. Schenectady, NY, 1998.

CERTIFICATIONS:

New York State Department of Environmental Conservation certified data validator for Organic and Inorganic analysis data summary reports.

JASON M. HOMERIGHAUS: LAB MANAGER

PROFESSIONAL EXPERIENCE:

2003-2005 **Managing Director/Technical Director**

Friend Laboratory, Inc.
Waverly, NY

-Responsible for managing all aspects of a full-service NYSELAP laboratory as senior manger.
-Responsible for staff of 22 employees and 4 managers, including all staffing decisions
-Management of $2 million annual operating budget and all contract negotiation/acceptance
-Oversee all technical and quality assurance operations as Technical Director, including method development, analysis/instrument troubleshooting, and technical data review

2002 –2003 **Business Manager**

English Sewage Disposal, Inc.,
Elmer, NJ

-Business management for septic, portable toilet and industrial services company of 15 employees and $2,000,000 in annual sales
-Oversaw all trucking, maintenance, service, financial, compliance and customer service operations
-Responsible for special project sales, field management and site supervision
-Managed marketing program; conducted sales and training for new customer development

2000–2002 **Laboratory Manager**

South Jersey Testing Laboratory
Bridgeton, NJ, June

-Responsible for the management of NJ certified environmental laboratory with a $750,000 annual budget and a staff of 10. Also responsible for all technical, customer service, purchasing, budgets and financial management

1999-2000 **Laboratory/Production Manager,**

The C.R. Warner Companies
Philadelphia, PA,

-Managed all laboratory, transfer, yard and processing activities for waste oil recycling operation while also responsible for all regulatory testing, hazardous waste handling, budgets, supplies and inventory

1996-1999 **Regulatory Compliance Officer**

East Norriton-Plymouth-Whitpain Joint Sewer Authority (Norristown, PA, June)
-Responsible for all regulatory testing, hazardous waste handling, MIPP program management, DMR compliance and plant process control monitoring & adjustment.

EDUCATION:

1996 Bachelor of Science in Biochemistry,
State University of New York at Oswego

TRAINING:
-OSHA 40-hour course
-SAC I & II (Wastewater Treatment Plant Operator Training)
-Chlorine use and handling
-Confined space entry
-National Association of Wastewater Transporters (NAWT) Onsite Treatment Training

JASON M. HOMERIGHAUS: LAB MANAGER

-PSEG Nuclear, Onsite Access Training

LICENSES:
-PA SB-1 Waste Water Treatment Plant Operators License
-Class A CDL with Air Brake and Tank Certifications
-NAWT Inspection Certification #NA13823WT for onsite treatment systems
-Previously approved for un-escorted access at PSEG Salem Nuclear Power Station

***Association Memberships**:*

-American Water Works Association (AWWA)
-American Association of Cereal Chemists (AACC)
-New York State Association for Solid Waste Management (NYSASWM)
-Eastern Pennsylvania Water Pollution Control Operators Association (EPWPCOA)
-Independent Oil and Gas Association of NY (IOGA)

INGA C. HOTALING: ORGANICS ANALYSIS DEPARTMENT MANAGER

PROFESSIONAL EXPERIENCE:

2001-Present: **GC Section Manager/Chromatography Systems Manager**
Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, New York 12308

Responsible for management of the GC analysis section including analysis scheduling, analyst training and instrument maintenance Performs High Resolution PCB congener analysis, data processing and reporting for a variety of matrices, Responsible for Laboratory Chromatography Database System management. Duties include system configuration ,optimization and maintenance.

1997-2001: **Chemist/Chromatography Systems Manager**
Northeast Analytical, Inc.
301 Nott Street/2190 Technology Drive
Schenectady, New York 12305

Performs High Resolution PCB congener analysis, data processing and reporting for a variety of matrices,. Responsible for Laboratory Chromatography Database System management. Duties include system configuration ,optimization and maintenance.

1993-1997: **Chemist/Silicone Study Manager**
Northeast Analytical, Inc.
301 Nott Street
Schenectady, New York 12305

Responsible for analysis of Polydimethylsiloxane and Dimethylsilanediol in various sample matrices using ICP-SEP/ICP-HPLC. Duties in this field include scheduling sample preparation and analysis, method development, data evaluation and processing, and instrument maintenance and troubleshooting. Duties in other areas include High Resolution PCB congener analysis, data processing, and chromatography workstation optimization for congener specific PCB analysis.

1991-1993: **Chemist**
Northeast Analytical, Inc.
301 Nott Street
Schenectady, New York 12305

Responsibilities included extraction and purification of diverse sample matrices for High Resolution PCB analysis. Data processing for PCB screens on Megabore and 8080 PCB analysis. Sample extract disposal and archival. Performed analysis for metals by FLAA and GFAA.

1989-1991: **Stallion Manager**
Highcliff Farm
Division of Tri-Noble Stable
Delanson, New York

Provided and maintained high quality care for thoroughbred breeding stallions and surroundings. Assisted with surgeries, semen evaluation, transtrachea wash, plasma transfusion, vaccination, x-rays, and administered medication.

1986-1989: **Broodmare Manager**
Schoenborn Brothers Farm
Climax, New York

Responsibilities included organization of daily work schedule, mare and foal care, sales preparation, and assistance in veterinarian reproductive work.

1986: **Chemical Laboratory Assistant**
Smith & Smith Environmental Laboratory
Sierra Vista, Arizona

INGA C. HOTALING: ORGANICS ANALYSIS DEPARTMENT MANAGER

Initiated GC procedure for testing of herbicides in soil and sludges. Completed bacteriological and chemical water analysis. Other duties included water sample collection and preparation.

1983-1985: **Chemical Laboratory Assistant**
National Agricultural
Research & Development Institute
Augustenberg, Germany

Planned and carried out analytical procedures in connection with several research grants which included studies of heavy metals in sewage sludge and their effect in agriculture. Uptake of heavy metals by crops in relation to their concentration in the soil solution. Determination of pesticides and PCBs in municipal waste waters and the uptake of strontium-90 in agricultural crops in connection to heavy metal content and soil type.

EDUCATION:

1983: Karl-Engler School
National Agricultural
Research & Development Institute
Augustenberg, Germany

PUBLICATIONS:

Contributing editor to *Guide to Environmental Analytical Methods* 4th ed. A summary of Quality Assurance requirements and other pertinent information for select analytical methodologies. Published by Genium Publising Corp. Schenectady, NY, 1998.

Casey, A.C., Wagner R.E., Hotaling I.C., and Carlson H. (2005). Application of Automated Solid Phase Extraction for Determination of Polychlorinated Biphenyls (PCB) in Water. Presented before the Annual Conference on Soils, Sediments and Water University of Massachusetts at Amherst

Casey, A.C., Wagner R.E., Hotaling I.C., and Carlson H. (2006). Application of Automated Solid Phase Extraction for Determination of Polychlorinated Biphenyls (PCB) in Water Presented with Horizon Technology at PITTCON 2006

ANTHONY J. MAIELLO: ORGANICS ANALYSIS ANALYST

PROFESSIONAL EXPERIENCE:

1997-Present: **GC Analyst -8082 & 8081 Specialist Organics Section**
Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, New York 12305

Responsible for analyzing tissue, water, oil, wipes and soil/sediment samples for pesticide, PCB Aroclors and herbicide contaminants. Performs EPA SW846-8081, SW846-8082, SW846-8015 and 608 GC/ECD analyses daily.

1990-1997: **Senior Chemist**
Friend Laboratory, Inc.
Environmental Laboratory Services
Waverly, New York

Senior chemist in the Inorganic Chemistry Section. Responsible for all Wet Chemical Miscellaneous Testing and Instruments Testing on Pharmaceuticals. Performed EPA and Standard Methods Testing on Drinking Water, Waste Water, and Soils for chemical species requested. Installed Technicon ™ Autochemistry Systems for Phenols, Cynaide, and Phosphates with Computer Data Acquisition. Set up system and performed EPA 531.1 for Carbamate Pesticides and EPA 547 Glyphosate in Drinking water by HPLC. Promoted to Manager of Wet Chemistry Department on 6/93.

1987-1990: **Staff Chemist**
Manpower Technical Services
At Imaging and Sensing Technology Corp. Horseheads, NY
New York State Electric and Gas Binghamton, NY

Technical Services Laboratory:

Worked on FT-IR of organic materials used in manufacturing; Emission Spectroscopy of metals and organic materials; GC analysis of waste water for volatile organics, ICP analysis of metals in waste water, Impurities in alloys and quantification of major elements in alloys.

NYSEG- Environmental Laboratory:

Prepared samples for semi-volatiles analysis. Prepared and analyzed samples for GC analysis for pesticides in water. Analyzed anions by ion Chromatography.

1981-1987 **Norwich-Eaton Pharmaceuticals Inc. Staff Scientist**
Norwich, NY

Began as Research Scientist II in the Analytical Services Section. Worked with experimental dosage forms, bulk drugs. and special projects as assigned. Consultant for medical device testing programs. Modified existing methods through an evaluation program; adapted methods to new dosages forms or to competitor's products. Promoted to RS III (Staff Scientist) 5/85. Reviewed and witnessed data generated by technicians. Maintained compliance with GLP and FDA guidelines.

1976-1980 **QA Analytical Chemist**
USCI Div. of C.R. Bard Inc.
(Medical Device Manufacturer)
Glens Falls, NY

Worked as chemist and supervisor of a small industrial QA laboratory. Worked on chemical and physical test methods. Responsible for developing testing and specifications for raw materials, in-process mixes, and finished products.

1969-1976 **QC Chemist**
Norwich-Eaton Pharmaceuticals (Norwich, NY)

ANTHONY J. MAIELLO: ORGANICS ANALYSIS ANALYST

Started as Jr. Chemist working with wet chemical and instrumental techniques on raw materials vs. USP/NF compendia and in-house specifications. Promoted to Control Chemist 8/73 and assumed responsibility for all GC testing for the QC laboratory. Assumed responsibility for calibration and maintenance of QC lab instruments in 1974.

CERTIFICATIONS AND CONTINUING STUDIES:

Gas Chromatography: Sadtler Research Laboratories, Philadelphia, PA
Liquid Chromatography: Waters Associates, Milford, MA.
HPLC Updates and Advances: Dr. Lloyd R. Snyder, LC Resource Inc. (in-house course), San Jose, CA.
Thermoanalytical Methods: Center for Professional Advancement, East Brunswick, N.J.
Quality Assurance Principles: Rensselaer Polytechnic Institute, Troy, N.Y.

Professional Affiliations

Member: The American Chemical Society
American Chemical Society Short Course in Interpretation of Mass Spectra

MARSH QIAN: INORGANICS DEPARTMENT MANAGER

PROFESSIONAL EXPERIENCE:

11/05-PRESENT: **Inorganics Lab Manager**
Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, New York 12308

Inorganics Lab Manager of Northeast Analytical, Inc.; supervise and manager laboratory staff in the chemical analysis of various environmental samples for metal analysis by ICP, GFAA and CVA; amend and implement analytical methods and procedures that are currently being used; responsible for the review of data analyzed by the Inorganic department; maintain laboratory certification programs; maintain and instruct laboratory personnel on laboratory instruments; responsible for the Ward packaging system.

11/04-11/05 **Chemist and Department Supervisor**
Mid-Atlantic Environmental Labs, Inc.
Claymont, DE

In charge of the daily activities of metal department, including but not limited to sample analysis, QC, data validation and packaging, instrumentation (ICP, ICP/MS, GFAA, CV, GC/MS, UV-VIS, and etc), various software practice, technician training, documentation, audit and certificate response, method development and validation, SOP revise and updating

10/00-11/04 **Senior Chemist and Environmental Lab Manager**
Kuo Testing Labs, Inc.
Othello, WA

- Kept good lab management and instrument maintenance, supervised and trained technicians, troubleshoot and solved problems.
- Good at instruments of ICP, FLAA, GFAA, UV-VIS, N&S Analyzer, FIA, GC, ISP, titration and other wet chemistry procedures.
- Increased 30 % of business by providing better Quality Control (QC) and Quality Assay (QA) practices, fast and accurate results turning out and custom responses.
- Developed new testing methods and revised SOPs.
- Improved the state certificate program results from Accept to Excellent.
- Accomplished analysis of more than 10,000 samples yearly from industrial, agricultural, environmental, consulting and individual customs.
- Cut lab cost by working more efficiently.

9/98-10/00 **Research Assistant**
University of HI
Honolulu, HI

- Finished a two-year research project independently.
- Designed, setup and monitored laboratory bioreactors for conducting water treatment and evaluated the efficiency with various wet chemistry and instrument analysis.
- Accomplished experimental design, water treatment and quality analyses, data processing, and reports preparation.

10//89-9/98 **Senior Chemist, R&D Group Leader**
China Research Institute of Daily Chemical Industry
Shanxi, China

Organic synthesis, catalysts, wet chemistry and instrument analysis, formulation, industrial application, QC/QA, and etc.

EDUCATION:

2001 **M.S. Biosystems Engineering (Environmental)**
University of Hawaii, Honolulu, HI

MARSH QIAN: INORGANICS DEPARTMENT MANAGER

1989	**M.S. Organic Synthesis** China Research Institute of Daily Chemical Industry Shanxi, China
1986	**B.S. Chemistry,** Beijing Technology and Business University, Beijing, China

Publications

Qian, X., Yang, P. Y. and Maekawa, T. (2001) Potential application of EMMC technology for direct removal of nitrate from contaminated-groundwater. *ASIAN WATERQUAL 2001. Asia-Pacific Regional Conference*. Sept., Fukuoka, Japan

Qian, X., Yang, P. Y. and Maekawa, T. (2000) Evaluation of Direct Removal of Nitrate with EMMC Technology Using Ethanol as Carbon Source. *Water Environment Research,* 2001, **73**(5), pp 584-589

Qian, X (2001). EMMC for Land-Limited Biological Denitrification for Nitrate-Contaminated Groundwater. *Master Thesis*. University of Hawaii at Manoa, Honolulu, HI, USA

Qian Xia (2000). EMMC for Land-Limited Biological Denitrification for Nitrate-Contaminated Groundwater. *Poster presentation to the Hawaii Water Environment Association 22nd Annual Conference*. June, Honolulu, HI, USA

Qian, X. and Yang, P.Y. (2000). EMMC for Land-Limited Biological Denitrification for Nitrate-Contaminated Groundwater. *Poster presentation to the 12th Annual CTAHR Student Research Symposium.* April, University of Hawaii at Manoa, Honolulu, HI, USA

Qian, X. and Yang, P.Y. (1999). EMMC Technology for Nitrate Removal from Contaminated Groundwater. *Poster presentation to 11th Annual CTAHR Student Research Symposium*. April, University of Hawaii at Manoa, Honolulu, HI, USA

Qian, X.and Yang, P. Y. (1999). Land-Limited Biological Denitrification for Nitrate contaminated Groundwater for Water Supplies. *Research Reports*. 1998~1999.

Qian, X. and Zhang, G. Y. (1997). Theory & Practice of Dehydroamination of Fatty Alcohol in a Fixed-Bed Reactor. *Riyong Huaxue Gongye*, (2), 4-8 (Ch)

Qian, X.(1997). Preparation of Fatty Alcohols and Propane diol by Direct Hydrogenation of Fats/Oils. *Riyong Huaxue Gongye*, (4), 31-34 (Ch)

Guo, J. G., Zhou, G. S, Tang, L. S, Qian, X. and Cheng, N. (1997). Study on the Preparation of Aliphatic Alcohols by Catalytic Hydrogenation of Coconut Acid Methyl Esters. *Riyong Huaxue Gongye*, (5), 1-3 (Ch)

Zhou, G., Guo, J. G., Qian, X. and Tang, L. S. (1997) Development of Catalysts for Hydrogenation of Coconut methyl Ester to Fatty Alcohols under Mid-Pressure. *Riyong Huaxue Gongye*, (3), 7-9 (Ch)

Qian, X.(1996) Process and Catalysts of Hydrogenating Fatty Acid Methyl Esters to Alcohols under Mid-Pressure. *Detergent & Cosmetics*, (2), 3-4, 36 (Ch)

"Fatty Acid in Industry Press--- process, properties, derivatives, applications.", 1992, China Light Industry Press, translator of partial sections

MICHAEL GLENN: ORGANICS EXTRACTION OPERATIONS MANAGER

PROFESSIONAL EXPREIENCE:

2006 – Present: **Extractions Laboratory Manager**
Northeast Analytical Inc.
2190 Technology Drive
Schenectady, NY 12308

Responsible for the direction and supervision of the day shift staff for environmental extractions. Train, supervises and manages the extraction technicians involved in the extraction of soil, sediment, tissue and water samples for PCB, pesticides, herbicides, and semi-volatile analysis. Responsibility included coordinating with the analytical group to ensure that samples were extracted properly.

2005-2006: **GC Analyst**
Northeast Analytical
2190 Technology Drive
Schenectady, NY 12308

Analyze PCBs via EPA method 8082: Aroclor pattern, in various matrices using GC/ECD, analyze pesticide and human serum. Prepare sample dilutions and calibrations standards. Analyze chromatograms; assemble data packages for clients of results processed.

2003 – 2005 **Non-Volatile Organics Extraction Supervisor**
Northeast Analytical Inc.
2190 Technology Drive
Schenectady, NY 12308

Responsible for the direction and supervision of the day shift staff for environmental extractions. Train, supervises and manages the extraction technicians involved in the extraction of soil, sediment, tissue and water samples for PCB, pesticides, herbicides, and semi-volatile analysis. Responsibility included coordinating with the analytical group to ensure that samples were extracted properly.

2003 **Non-Volatile Organics Extraction**
Northeast Analytical Inc
2190 Technology Drive
Schenectady, NY 12308

Performed extraction of soil, sediment, tissue and water samples for PCB, pesticides, herbicides, and semi-volatile analysis.

EDUCATION: **Western New England College**
Bachelor of Science Degree - May 2003
Major: Chemistry
Minors: Education & Psychology

PRESENTATIONS:

Casey, A.C., Wagner R.E., Herold T. E. and Glenn M. (2006) The Evaluation of Extraction and Cleanup Methods for the Determination of PCB Aroclors in Caulking and Sealing Material. Presented before the Annual Conference on Soils, Sediments and Water University of Massachusetts at Amherst

ROY SMITH, JR. : SEMI-VOLATILES DEPARTMENT MANAGER

Professional Experience:

May 2004 – Present: **High Res. PCB Congener and SVOC Supervisor**
Northeast Analytical
2190 Technology Drive
Schenectady, NY 12308

Responsible for high resolution PCB Congener and semi-volatile analysis of environmental samples of varying matrices, including soil, oil, water and tissue. Analysis of PCB's is by two specialized types of analysis known as CQCS (Comprehensive Quantitative Congener Specific Analysis) and EPA Method 680. Also involved in organics analysis, data interpretation and sample and report review. Other responsibilities include the maintenance and upkeep of the equipment and QA/QC compliance.

June 1999-May 2004: **Volatiles Supervisor and Asst. Lab. Director**
American Westech, Inc.
Harrisburg, PA 17112

Responsibilities included the supervision and management of the organic department. Additional responsibilities included; project management, analysis, data review and packaging, data and report review and monitoring for QA/QC compliance.

Nov. 1996-June 1999: **Laboratory Supervisor**
ChemSpec Analytical Laboratories
Harrisburg, PA 17112

Responsibilities included the supervision and management of daily operation of the laboratory from sample log-in to final report generation.

May 1990-Sept. 1996: **Environmental Scientist**
Centre Analytical Laboratories
State College, PA 16801

Responsible for analysis of volatile environmental samples by GC and GC/MS. Other duties included TOX and Oil and Grease analysis as well as TCLP preparation.

EDUCATION:

May 1990: **B.S. Chemistry**
Juniata College
Huntingdon, PA

ANDREA C. MASWICH: VOLATILES DEPARTMENT SUPERVISOR

PROFESSIONAL EXPERIENCE:

January 2005-Present: GC/GCMS Volatiles Supervisor
Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, NY 12308

Responsible for the supervision of sample analysis for volatile compounds by GC and GCMS. This includes the following: reviewing all data reported to clients for accuracy and EPA method compliance, maintaining all instruments by performing routine and non-routine maintenance, training new analysts, and reviewing all SOPs for accuracy and updates.

August 2002-January 2005: GC/GCMS Volatiles Analyst
Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, NY 12308

Responsible for water and soil samples analysis for volatile compounds by GC/GCMS. This includes the following: reporting final data, calibrating the GC/GCMS, diluting samples to ensure that compounds are within range of the instrument, disposing of samples, and maintaining equipment; GC/GCMS, purge and trap concentrator, and autosampler.

May 2001-August 2002: Lab Technician
Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, NY 12308

Responsible for the extractions of soil, sediment, tissue and water samples for PCB, pesticides, and semi-volatile analysis; via soxhlet, Automated Solvent Extraction, continuous liquid liquid extraction, and seperatory funnel as well as their clean up procedures.

EDUCATION:

2000: B.A. Earth and Atmospheric Sciences
State University of New York at Albany
Albany, New York

APPENDIX D

EMPLOYEE TRAINING LOG

COPLANAR PCB METHOD	INITS.	DATE	TRAINER'S INITS.	DATE	SUPERVISOR'S INITS.	DATE
Reagent Prep						
Silica Gel Activation/Deactivation						
Silica Gel Calibration						
Standards Prep						
Surrogates (PBB) 34/34 TBB & 24/4 DBB						
Calibration Curve Solutions						
Coplanar WHO Mix and NEA Congener Mix						
Spike Solutions						
Column Chromatography						
Silica Gel Clean-Up Columns						
Solvent Reduction						
TurboVap						
MicroBlow Down						
Clean-Ups						
Acid Wash						
Mercury Shakes						
HPLC Operation and Maintenance						
Gilson Pump Operation						
Gilson Pump Cleaning and Maintenance						
Waters Autosampler Operation						
Waters Autosampler Cleaning and Maintenance						
Waters UV Detector Operation						
Waters UV Detector Tuning and Calibration						
Waters UV Detector Cleaning and Maintenance						
Coplanar GC Method						
Set-up of GC Method						
Calibration						
Chromatogram Review and Edits						
Millennium Coplanar Reports						
Coplanar GC/MS Method						
Set-up of Method						
Calibration						

APPENDIX E

NEA CHAIN OF CUSTODY DOCUMENT EXAMPLE

CHAIN OF CUSTODY RECORD PAGE _____ OF _____

NORTHEAST ANALYTICAL, INC.
2190 Technology Drive, Schenectady, NY 12308
Telephone (518) 346-4592 Fax (518) 381-6055
www.nealab.com information @nealab.com

LRF # _____
(NEA USE ONLY)

DISPOSAL REQUIREMENTS: (To be filled in by Client)
- ◯ RETURN TO CLIENT
- ◯ DISPOSAL BY NORTHEAST ANALYTICAL
- ◯ ARCHIVAL BY NORTHEAST ANALYTICAL

Additional charges incurred for disposal (if hazardous) or archival. Call for details.

CLIENT (REPORTS TO BE SENT TO):	**PROJECT#/PROJECT NAME:**
PROJECT MANAGER:	**PROJECT LOCATION (CITY/STATE) ADDRESS:**
PHONE:	
SAMPLED BY: (Please Print)	**REQUIRED TURN AROUND TIME:**
SAMPLING FIRM:	**NAME OF COURIER (IF USED):**
	Data Report: ☐ CLP* ☐ Certificates Only
ELECTRONIC RESULTS FORMAT: .PDF ☐ EXCEL (.CSV) ☐	E-MAIL ADDRESS:
FAXED RESULTS ☐	FAX #:

ENTER ANALYSIS AND METHOD NUMBER REQUESTED

PRESERVATIVE CODE:
BOTTLE TYPE:
BOTTLE SIZE:
NUMBER OF CONTAINERS

PRESERVATIVE KEY
0 - NONE
1 - HCL
2 - HNO3
3 - H2SO4
4 - NaOH
5 - Zn. Acetate
6 - MeOH
7 - NaHSO4
8 - Other _____

SAMPLE ID	DATE	TIME	MATRIX	GRAB/ COMP	LAB SAMPLE ID (NEA USE ONLY)	REMARKS:

AMBIENT OR CHILLED: TEMP: COC TAPE: Y N PROPERLY PRESERVED: Y N OTHER NOTES:
RECEIVED BROKEN OR LEAKING: Y N COC DISCREPANCIES: Y N RECVD W/I HOLDING TIMES: Y N

RELINQUISHED BY	RECEIVED BY	RELINQUISHED BY	RECEIVED BY	RELINQUISHED BY	RECEIVED BY
SIGNATURE	SIGNATURE	SIGNATURE	SIGNATURE	SIGNATURE	SIGNATURE
PRINTED NAME	PRINTED NAME	PRINTED NAME	PRINTED NAME	PRINTED NAME	PRINTED NAME
COMPANY	COMPANY	COMPANY	COMPANY	COMPANY	COMPANY
DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME

* **CLP LIKE DATA PACKAGE ADDITIONAL COST**

APPENDIX F

NEA INTERNAL CHAIN OF CUSTODY DOCUMENT

Northeast Analytical Inc.
2190 Technology Drive
Schenectady, NY 12308

Internal Chain of Custody Transfer

Sample Delivery Group: _____

Department _____

Received at Lab: Date: _____ Time: _____

	Received from:	Date:	Time:	Relinquished to:	Comments: Sample Location / reason for transfer / procedures
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					

Form: S:\FORMS\CATB\IN_COC_sample_and_extract.XLS (rev1, 7/23/2007)

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APPENDIX G

NEA INSTRUMENTATION LISTING

APPENDIX G LABORATORY INSTRUMENTATION

Northeast Analytical, Inc. utilizes state-of-the-art instrumentation. Following is a listing of major instrumentation at NEA:

Organics Analysis (non-Volatile):

- Agilent 6890N Gas Chromatograph equipped with 7683 Autosampler (100 position tray), micro electron capture detector, Split/Splitless injector. (Purchased 2001)
- Agilent 6890N Gas Chromatograph equipped with 7683 Autosampler (100 position tray), micro electron capture detector, Split/Splitless injector. (Purchased 2001)
- Agilent 6890 Gas Chromatograph equipped with 7683 Autosampler (100 position tray), micro electron capture detector, Split/Splitless injector. (Purchased 2001)
- Varian 3800 Gas Chromatograph equipped with LEAPS autosampler with PAC System Technologies (216 positions), Dual Ni-63 electron capture detector universal capillary 1177 Split/Splitless injector. (Purchased 2002)
- Varian 3800 Gas Chromatograph equipped with LEAPS autosampler with PAC Syestem Technologies (216 positions), Dual Ni-63 electron capture detector universal capillary 1177 Split/Splitless injector. (Purchased 2002)
- Varian 3800 Gas Chromatograph equipped with Varian CP-8400 Autosampler (48 position), Ni-63 electron capture detector universal capillary 1177 Split/Splitless injector. (Purchased 2002)
- Varian 3800 Gas Chromatograph equipped with Varian 8200 Autosampler (48 position), Ni-63 electron capture detector universal capillary 1079 injector. (Purchased 1999)
- Varian 3800 Gas Chromatograph equipped with Varian 8200 Autosampler (48 position), Ni-63 electron capture detector universal capillary 1079 injector. (Purchased 1999)
- Varian 3400 Gas Chromatograph equipped with Varian 8100 Autosampler (48 position), Ni-63 electron capture detector, Split/Splitless injector and universal injector. (Purchased 1991)
- Varian 3400 Gas Chromatograph equipped with Varian 8100 Autosampler (48 position), Ni-63 electron capture detector, Split/Splitless injector and universal injector. (Purchased 1990)
- Varian 3400 Gas Chromatograph equipped with Varian 8035 Autosampler (60 position), FID detector, Split/Splitless injector and universal injector. (Purchased 1990)
- Varian 3400 Gas Chromatograph equipped with Varian 8100 Autosampler (48 position), Ni-63 electron capture detector, SPI injector and universal injector. (Purchased 1989)
- Varian 3400 Gas Chromatograph equipped with Varian 8100 Autosampler (48 position), Ni-63 electron capture detector, SPI injector and universal injector. (Purchased 1989)

Volatile Organics Analysis (VOA):

- Varian Saturn 2000 GC/MS with Varian GC 3900. (Purchased 2003)
    - Tekmar 3000 Purge and Trap Concentrator. (Purchased 2001)
    - Varian Archon 51 position zero headspace robotic autosampler equipped with magnetic stir bar accessory for closed system volatiles analysis (SW-846 Method 5035). (Purchased 2002)
- Varian Saturn 2200 GC/MS with Varian GC 3800. (Purchased 2003)
    - OI Analytical 4660 Eclipse Purge and Trap Concentrator (Purchased 2006)

        - Varian Archon 51 position zero headspace robotic autosampler equipped with magnetic stir bar accessory for closed system volatiles analysis (SW-846 Method 5035). (Purchased 2003)

- Varian 3400 Gas Chromatograph equipped with PID detector, ELCD detector, cryogenic controlled oven, and two universal injectors. (Purchased 1995)
    - OI Analytical 4660 Eclipse Purge and Trap Concentrator (Purchased 2006)
    - Varian Archon 51 position zero headspace robotic autosampler equipped with magnetic stir bar accessory for closed system volatiles analysis (SW-846 Method 5035). (Purchased 2003)

- Varian 3400 Gas Chromatograph equipped with FID detector, cryogenic controlled oven, and two universal injectors. (Purchased 1995)
    - Tekmar LSC 2000 Purge and Trap Concentrator (Purchased 1995)
    - Tekmar ALS 2016, 16 stage Purge and Trap Autosampler (Purchased 1995)

- Varian 3400 Gas Chromatograph equipped with FID detector, cryogenic controlled oven, and two universal injectors. (Purchased 1995)
    - Varian 8035 Autosampler

Semi-Volatile Organics Analysis (SVOA):

- Varian Saturn II GC/MS with Varian 3400 GC, NIST Spectral Library, 8100 Autosampler (48 position, low level), Chemical Ionization option. (Purchased 1993)

- Varian Saturn 2000 GC (with MS/MS option) with Varian GC 3800 (Model # 1177 and 1079 injectors), NIST Spectral Library, equipped with Varian 8200 Autosampler (48 position, low level). (Purchased 1999)

- Varian Saturn 2200 GC (with MS/MS and EI/CI capability options) with Varian GC 3800 (Model # 1079 injector), NIST Spectral Library, equipped with Varian 8200 Autosampler (48 position, low level). (Purchased 2003)

Inorganics Analysis:

- Spectro Ciros (Model # FCEM-1) Coupled Plasma (ICP) Atomic Emission Spectrometer with multi-configuration robotic Autosampler. (Purchased 2004)

- Varian SpectrAA-240Z Zeeman Automatic Absorption Spectrometer, with GTA120 Graphite Tube Atomizer and PSD120 Programmable Sample Dispenser and Super lamp accessories. (Purchased 2004)

- Leeman Hydra AA Mercury Analyzer, cold vapor atomic absorption. (Purchased 2001)

- Shimadzu TOC-V_{CSH} Total Organic Carbon Analyzer, with ASI-V Auto Sample Injector and SSM-5000A Solid Sample Module

- Tekmar-Dohrmann Total Organic Carbon (TOC) analyzer DC-190, with Tekmar-Dohrmann TOC Boat Sampler Model 183

- Buck Scientific Model 404, Infrared Wavelength Total Hydrocarbon Analyzer.

- Bausch & Lomb Spectronic 20 Visible wavelength spectrometer.

- Orion 720A pH Meter

Sample Digestion/Processing Equipment (Inorganics):

- Environmental Express Hot Block Digestion Unit with 36 positions (purchased 2004)

- Toxic Characteristic Leaching Procedure (TCLP) and Zero Headspace Extraction (ZHE) Apparatus.

Sample Extraction/Processing Equipment (Organics):

- J_2 Scientific AccuPrep GPC (Gel permeation Chromatography) system. (Purchased 2004)
- Dionex ASE 200 Accelerated Solvent Extractor with 24 position robotic autosampling for non-volatile organics extractions (4 units). (Purchased 1997-1998)
- Corning Accelerated One-Step Continuous Liquid/Liquid Extractor apparatus for aqueous non-volatile organic samples.
- Zymark Turbovap™ (3 units), 6-position solvent extract concentration device.
- Zymark Turbovap™ LV (1 unit), 24-position solvent extract concentration device.
- HEAT Systems™ Ultrasonic Extraction Apparatus.
- Horizon SPE-DEX 4790 Series Automated Solid Phase Extractor for non-volatile organics extractions (8 units). (Purchased 2001-2003)
- Soxhlet Extraction Apparatus for non-volatile organics samples (24 positions).

Miscellaneous Equipment:

- Metler AG204 Analytical Balance with 0.1 mg resolution equipped with serial interface for online data acquisition to LIMS (2 units).
- Metler PL303 Balance (2 units).
- Metler PJ-3600 Balance (1 unit).
- Fisher Model 200 Oven.
- VWR Model 1370GD Oven.
- VWR Model 1370FO Oven.
- Thermolyne Model 30400 Furnace (2 units).
- Miele Melabor G7783 Laboratory Dishwasher.

Data Processing Equipment:

- Local Area Network (LAN) including Main Novell Netware™ Fileserver and Millennium 32™ Chromatography Database Server.
- Thirty five network client workstations, stand alone and interfaced to analytical instrumentation.
- Waters Millennium 32 V3.05 Chromatography Workstation for gas chromatograph data management, each quipped with PC, interfaced to Chromatography Database Server.
- Waters EMPOWER Chromatography Workstations for gas chromatograph data management, each equipped with PC, interfaced to Chromatography Database Server.
- Varian Saturn II Data Acquisition/Processing Software/Target Thruput Processing Software
- Local Area Network Fileserver based tape backup and archival systems including 3-5Gbyte 4mm DAT tape drives managed by Seagate Storage Manager™ backup software.
- NEA™ LIMS (Laboratory Information Management System)

Miscellaneous Equipment:

- Metler AG204 Analytical Balance with 0.1 mg resolution equipped with serial interface for online data acquisition to LIMS (2 units).
- Metler PL303 Balance (2 units).
- Metler PJ-3600 Balance (1 unit).
- Fisher Model 200 Oven.
- VWR Model 1370GD Oven.
- VWR Model 1370FO Oven.
- Thermolyne Model 30400 Furnace (2 units).
- Miele Melabor G7783 Laboratory Dishwasher.

Sample Storage Equipment:

- Walk-in cooler (4ºC) – 12’ X 17’
- Walk-in freezer (-20ºC) – 10’ X 14’
- Thirteen various refrigerators for sample and extract storage.

APPENDIX H

INSTRUMENT RUN LOGBOOK EXAMPLE

NORTHEAST ANALYTICAL, INC. GCMS 06 INJECTION LOG

	NEA Sample ID	Saturn File ID	Date Analyzed	DILUTION	Saturn Quant. File	Comments
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

BFB:_____

8021 Indy:_____

CS2:_____ MTBE:_____

Ketones:_____ Surr/IS:_____

Gases:_____ Curve:_____

APPENDIX I

INSTRUMENT MAINTENANCE LOGBOOK EXAMPLE

NORTHEAST ANALYTICAL, INC. GC12 MAINTENANCE LOG

Instrument: GC-12 OI 4660 concentrator: A344466384P PAGE 30

Model Number: Varain 3400 Varian Archon Autosampler: Serial # 1365 Date: _____

Serial # 5919 Analyst: _____

VARIAN 800-926-3000

Reason for Maintenance: routine ☐ problem ☐

Routine maintenance performed

clipped column	☐	new column length	☐
cleaned PID bulb	☐	changed/cleaned sparge tube	☐
replaced Ni tube	☐	changed trap	☐
backflush Teflon transfer line	☐	changed transfer line	☐
replaced ion resin	☐	changed soil transfer line	☐
replaced column	☐	cleaned syringe barrel	☐
changed helium tank	☐	other	☐
changed HC trap	☐	other	☐
	☐	other	☐

PLEASE LIST ALL PARTS CHANGED

	PART NAME	PART NUMBER	VENDOR
1.)			
2.)			
3.)			
4.)			
5.)			
6.)			
7.)			
8.)			
9.)			

DESCRIBE PROBLEM OR NON-ROUTINE MAINTENANCE:

Problem resolution: (Include if and how problem was resolved)

S:\FORMS\LOG\VOA\[GC12m.]

APPENDIX J

NEA ANALYTICAL METHOD LISTING

APPENDIX J SAMPLE EXTRACTION AND PREPARATION METHODS

EXTRACTION METHODS: SOLID AND WATER

ORGANIC EXTRACTION METHODS:

3510c Separatory Funnel Extraction (Aqueous Non-Volatile Organics)
3520c Continuous Liquid Liquid Extraction (Aqueous Non-Volatile Organics)
3535 Solid Phase Extraction (SPE) (Aqueous Non-Volatile Organics)
3540c Soxhlet Extraction (Solid Samples - Non-Volatile Organics)
3545 Pressurized Fluid Extraction (Solid Samples Non-Volatile Organics) via Dionex® ASE
3550b Ultrasonic Extraction (Solid Samples- Non-Volatile Organics)
3580a Waste Dilution (Liquid Waste Samples- Non Volatile Organics)

INORGANICS (METALS) DIGESTION METHODS:

3005a Acid Digestion of Aqueous Samples for FLAA or ICP Spectroscopy
3010a Acid Digestion of Aqueous Samples for FLAA or ICP Spectroscopy
3020a Acid Digestion of Aqueous Samples for GFAA Spectroscopy
3050b Acid Digestion of Sediments, Sludges, Soils and Solids

VOLATILE ORGANICS EXTRACTION METHODS:
3585 Waste Dilution for Volatile Organics
5035 Closed System Purge and Trap and Extraction for VOA in soil and waste

ORGANIC ANALYSES: WATER

VOLATILE ORGANIC ANALYSIS BY GC
503.1 by EPA 502.2 Abbreviated List-Volatile Aromatic & Unsaturated Organic Compounds
602-Volatile Aromatic Compounds
8021-STARS-New York State DEC STARS List

VOLATILE ORGANIC ANALYSIS BY GC/MS
624-Purgeable Organics
8260b-Purgeable Organics by Capillary Column CLP Analyte list
8260b- Additional Charge for 10 Tentatively Identified Compounds (TICs)
8010-Volatile Halogenated Organic Compounds List by Method 8260
8020-Volatile Aromatic Compounds list by Method 8260

SEMI-VOLATILE ORGANIC ANALYSIS BY GC/MS
625-Base/Neutral & Acid Extractables Analyte List (no Pesticides or PCBs)

SEMI-VOLATILE ORGANIC ANALYSIS BY GC/MS
625-Base/Neutral Only Analyte List (no Pesticides or PCBs)
625-Acid Extractables Only Analyte List
PAHs by Method 8270
8270c-Base/Neutral & Acid Extraction CLP Analyte List
8270c-Base/Neutral Only CLP Analyte List
8270c-Acid Extractables Only CLP Analyte List
8270c-Stars NYS DEC Stars List
8270c-Additional Charge for 20 TICs

ORGANICS BY GC
508-PCBs by Aroclor
608-Pesticides & PCBs
608-PCBs by Aroclor
608-Pesticides
8151b-Herbicides (2,4-D and 2,4,5-TP)

8081a-Pesticides
8082-PCBs by Aroclor
8082-PCBs by Congener Specific

PETROLEUM IDENTIFICATION/QUANTITATION
8015 GRO-Total Petroleum Hydrocarbons as Gasoline
8015 DRO-Total Petroleum Hydrocarbons as Diesel
1664-Oil and Grease (Hexane Extractables)
NYS DOH 310-13-Petroleum Identification Quantification
NYS DOH 310-14-Petroleum Identification
NW-TPH by GC/MS (Oregon Method)

MISCELLANEOUS CHEMICAL ANALYSIS
415.2-TOC-Total Organic Carbon
415.1-POC-Particulate Organic Carbon
150.1-pH
1010-Flashpoint EPA Method 1010
Hardness EPA Method 200.7 by ICP Calcium and Magnesium.(calculation)
160.1-Total Dissolved Solids
160.2-Total Suspended Solids
160.3-Total Volatile Solids
160.4-Total Solids
Standard Methods 10200 H3-C Chlorophyll-a

ORGANIC ANALYSES: SOILS AND SOLIDS

VOLATILE ORGANICS ANALYSIS BY GC AND GC/MS
8010-Volatile Halogenated Organic Compounds List by Method 8260
8020-Volatile Aromatic Organic Compounds List by Method 8260
8021-STARS-New York State DEC STARS List by EPA 8260
8260-Volatile Organic Compounds CLP Analyte List
8260-Volatile Organic Compounds Extended (Full) Analyte List
8260-TIC (Tentatively Identified Compounds)
5035-Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples

SEMI-VOLATILE ORGANIC ANALYSIS BY GC/MS
8270c Base Neutral and Acid Extractables CLP Analyte List
8270c Base Neutrals Only CLP Analyte List
8270c Acid Extractable Only CLP Analyte List
8270c STARS
8100-PAH list by Method 8270c
8270c-20 TIC
8270d-Low Level PAHs in dust wipes

ORGANICS BY GC
8082-PCBs by Aroclor
8082-PCBs by Congener Specific
8081a-Pesticides
8082-PCBs in Oils
8082-PCBs in Wipes
8151-Herbicides (2,4-D and 2,4,5-TP)
PCB Wipe Kits (4oz Jar, Gauze, Hexane, and Template)

PETROLEUM IDENTIFICATION/QUANTITATION
8015 GRO-Total Petroleum Hydrocarbons as Gasoline
8015 DRO-Total Petroleum Hydrocarbons as Diesel
9071A-Oil and Grease
NYS DOH 310-13-Petroleum Identification Quantification

NYS DOH 310-14-Petroleum Identification

MISCELLANEOUS CHEMICAL ANALYSIS
TOC-Total Organic Carbon by Lloyd Kahn Method
9045-pH
9095A-Paint Filter Test
1020-Ignitability for Solids

ORGANIC ANALYSES: AIR SAMPLES

TO-4 PUF Filter Set-up*
TO-4 PUF Filter Set-up and Analysis by Method 8082
TO-10A Florisil Cartridge by Method 8082
TO-13A PUF and Analysis by Method 8270
NIOSH 5503-Florisil Cartridge by Method 8082

METALS ANALYSIS:

INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY (ICP)

The following elements are available by ICP EPA 6010b (solids and water) and EPA 200.7 (water)

Aluminum	Calcium	Lead	Selenium	Tin
Antimony	Cadmium	Magnesium	Silicon	Titanium
Arsenic	Chromium	Manganese	Silver	Vanadium
Barium	Cobalt	Molybdenum	Sodium	Zinc
Beryllium	Copper	Nickel	Strontium	Platinum
Boron	Iron	Potassium	Thallium	

EPA 6010c Low Level Lead in Dust Wipes

Sample Digestion/Preparation by Methods 3005A (Water) and 3050 (Solids)

GRAPHITE FURNACE ATOMIC ABSORPTION (GFAA)

The following elements are available by Graphite Furnace (GFAA) EPA 7000 series methods and EPA 200 series (Water)

Antimony	Cadmium	Lead	Selenium	Vanadium
Arsenic	Chromium	Manganese	Silver	
Beryllium	Copper	Nickel	Thallium	

Sample Digestion/Preparation by Methods 3005A (Water) and 3050 (Solids)

MERCURY by COLD VAPOR ATOMIC ABSORPTION (CVAA)
EPA Methods 7470(water), 7471(solids) and EPA 245.2 (water)

CHROMIUM, HEXAVALENT
Hexavalent Chromium, dissolved by Colorimetric Method (Diphenyl Carbazide) Method SM 18 3500-CrD

REGULATORY GROUPS
The following Regulatory Metals analyses are available as a group (Which includes Digestion):

ICP-Metals Scan (40 CFR Part 136, App C) (25 elements)
Appendix IX (24 Elements)
TCL-Target Compound List (23 Elements)
PPL-Priority Pollutant List (13 Elements)

REGULATORY GROUPS
RCRA-Appendix IX List (17 Elements)
NPDES List (23 Elements)
RCRA Hazardous Waste List (8 Elements)
Dissolved Metals Filtration

RCRA WASTE CHARACTERIZATION

TOXICITY CHARACTERISTIC LEACHING PROCEDURE
40 CFR 261.24 / SW846 Method 1311

EXTRACTIONS
1311-TCLP Extraction Volatiles (ZHE)
1311-TCLP Extraction Semi-Volatiles and Metals

ANALYSIS
TCLP-Metals (8 RCRA)
TCLP-Volatiles (8260b) List
TCLP-Semi-Volatiles (8270c) List
TCLP-Herbicides (8151b) List
TCLP-Pesticides (8081) List
9045-Corrosivity-pH
7.3-Reactivity
1010-Ignitability
TCLP 8021 Stars
TCLP 8270 Stars

CONGENER SPECIFIC PCB ANALYSIS:

EPA Method 8082 PCB Congeners with Comprehensive Quantitative Congeners Specific Analysis CQCS
Green Bay Mass Balance Method
EPA Method 680 PCB Homologs by GC/MS
Low Resolution GC/MS Congeners (In house Method)

PCB CONGENER LISTS

Green Bay Mass Balance Congener List:
Please contact the Lab for details and congeners reported by this method. This method relies on the use of Aroclor standards to report approximately 165 PCB congeners.

8082 Comprehensive Method Congener List:
This method reports all 209 PCB congeners, utilizing a mathematical deconvolution for PCB congeners that co-elute during GC analysis.

8082 WHO Coplanar Method Congener List:
This method reports all 12 Congeners listed by IUPAC Congener Number: 81, 77, 123, 118, 114, 105, 126, 167, 156, 157, 169, and 189 utilizing hypercarb fractionalization with ECD detection.

NOAA Congener List by National Status and Trends Programs listed by IUPAC Congener Number:
8,18, 28, 44, 52, 66, 77, 101, 105, 118, 126, 128, 138, 153, 170, 180, 187, 195, 206, & 209

USACE Congener List by US Army Corps of Engineers listed by IUPAC Congener Number:
8, 18, 28, 44, 49, 52, 66, 87, 101, 105, 118, 128, 138, 153, 170, 180, 183, 184, 187, 195, 206, & 209

Method 8082 Congener List by SW-846, Update III, 12/96 listed by IUPAC Congener Number:
1, 5, 18, 31, 44, 52, 66, 87, 101, 110, 138, 141, 151, 153, 170, 180, 183, 187, & 206

IUPAC Congener Number: The IUPAC Congener Number is a consecutive number system from 1 to 209 that labels all

209 possible PCB Congeners. The labeling sequence conforms to the International Union of Pure and Applied Chemistry (IUPAC) rules for nomenclature of organic chemistry.

REFERENCES:

Green Bay Mass Balance Congener List: Quality Assurance Plan, Green Bay Mass Balance Study, I. PCBs and Dieldrin, USEPA Great Lakes National Program Office, Final Draft, D.L. Swackhamer, December 11, 1987.

Frame, George M.; Cochran, Jack W.; Bowadt, Soren S.; "Complete PCB Congener Distributions for 17 Aroclor mixtures determined by 3 HRGC systems optimized for Comprehensive Quantitative Congener Specific Analysis," (1996), J. High Resolution Chromatography Volume 19: 657-668.

Frame, George M., "A Collaborative Study of 209 PCB Congeners and 6 Aroclors on 20 Different HRGC Columns 1. Retention and Coelution database," (1995), Fresenius J., Anal. Chem., 1141: 1-13.

NOAA Congener List: NOAA Technical Memorandum NOS ORCA 71, National Status and Trends Program for Marine Environmental Quality; Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992; Volume 1 Overview and Summary of Methods; G.G. Lauenstein and A.Y. Cantillo (Editors); Silver Spring, Maryland; July 1993.

USACE Congener List: Evaluation of Dredged Material Proposed for Ocean Disposal (Testing Manual) EPA/USACE, 1991. Also: USACE New York District, EPA Region II Guidance for Performing Tests on Dredges Material Proposed for Ocean Disposal, 1992.

Method 8082 Congener List: Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846), Third Edition, Final Update III, Dec 1996.

APPENDIX K

CORRECTIVE ACTION REPORT (CAR) EXAMPLE

NORTHEAST ANALYTICAL, INC.
2190 TECHNOLOGY DRIVE
SCHENECTADY, NY 12309

REPORT #: _____(QA Use Only)

DATE: _____

CORRECTIVE ACTION PLAN REPORT

I.) Identify Affected Samples (if applicable):

Sample Number(s) or LRF #(s):_____

Sample Matrix: Water [ ] Soil/Solid [ ] Biota [ ] Other: _____ (specify)

Type of Sample(s): QA/QC [ ], Client [ ], Proficiency Test [ ] Other: _____

Analysis and/or Preparation Method(s) _____ (provide Method # ID as applicable)

Laboratory SOP(s)_____ (list all applicable SOPs)

II.) Identify Problem (or Complaint) (To be completed immediately by individual that discovered problem)

Date Problem Discovered:_____ Desired Resolution Date: _____

Discovered by: _____ Name/Title/Company

Description of Problem:

III) CAP Team (The team responsible for the investigation of this issue)

Responsible Parties (include title)

IV) Problem Investigation (To be completed by individual(s) responsible for resolution of the problem)

Date(s)/Time(s) Problem(s) Occurred:

Personnel Involved:

Equipment Involved:

Location:

Probable or known causes of problem (why did problem occur):

Steps taken to investigate problem:

IV) Immediate (Interim) Corrective Actions:

Immediate (Interim) Corrective Action Initiated by:

_____ Name /Title Date: _____
Signature

V) Identification of Root Cause (factors) Contributing to the Problem

VI) Final Corrective action plan (CAP) (Include steps taken to prevent recurrence of problem):

Action #1		
Responsible Party:	**Plan Date:**	**Completion Date:**

Action #2		
Responsible Party:	**Plan Date:**	**Completion Date:**

Action #3		
Responsible Party:	**Plan Date:**	**Completion Date:**

VII) Quality Assurance, Monitoring and Review (**Detail any QA steps to be taken to ensure effectiveness of CAP)**

Monitoring and Review (Steps to be taken to ensure long term effectiveness of the CAP)

Monitoring Step #1		
Responsible Party:	**Plan Date:**	**Completion Date:**

Monitoring Step #2		
Responsible Party:	**Plan Date:**	**Completion Date:**

Monitoring Step #3		
Responsible Party:	**Plan Date:**	**Completion Date:**

VIII) Supervisor Review *(To be completed by supervisor responsible for project/area/function)*

Response and ICAP review: Acceptable [ ] Unacceptable [ ]

If unacceptable, preferred course of action:

Comments:

IX) Quality Assurance Officer Review *(To be completed by the Quality Assurance Officer)*

Response and ICAP review: Acceptable [X] Unacceptable [ ]

If unacceptable, preferred course of action:

Comments:

X) Management Review *(To be completed by Lab Director or other Manager)*

Response and ICAP review: Acceptable [ ] Unacceptable [ ]

If unacceptable, preferred course of action:

Comments:

XI) Final CAP Resolution and Closeout **(to be completed by the QAO and reviewed by management and QA during annual review)**

Targeted CAP Closeout Date:

Actual CAP Closeout Date:

Final CAP Review

Was CAP successful at addressing the issue?

Were long-term monitoring and review processes sufficient?

Is any additional follow-up required?

What were the Lessons Learned?

Follow-up recommendations:

Final distribution: Client Folder(s) [ ], QA-CAR folder [ ] Other [ ] (specify_____)

Q:\CAR Reports\2007.doc

APPENDIX L

LABORATORY METHOD BLANK FORM EXAMPLE

4A

Volatile Method Blank Summary

Northeast Analytical Inc.		SDG No.:	06090045
Lab File ID:	Z0611BM1	Lab Sample ID:	VMBLK01
Date Analyzed:	9/12/2006	Time Analyzed:	02:48
GC Column:	DB624, ID: 0.32 (mm)	Heated Purge:	No
Instrument ID:	GCMS1	NEA Form ID: epa-601.xls	
Method:	EPA-8260	NEA File ID: S:\FORMS\CATB\601\[EXAMPLE.XLS]4A (1)	

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, AND MSB

	CLIENT SAMPLE ID	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
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27				
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29				
30				

APPENDIX M
LABORATORY CONTROL SPIKE FORM EXAMPLE

FORM 3
Water Volatile Lab Control Spike Recovery

Northeast Analytical Inc. SDG.: 06090025

LCS Sample No.: AJ09876L NEA Form ID: S:\FORMS\CATB\EPA-601.XLS

NEA File ID: S:\FORMS\CATB\CLP-3D.XLS

Compound	SPIKE AMOUNT ADDED (µg/L)	LCS Concentration (µg/L)	LCS % REC	QC Limits REC.
Dichlorodifluoromethane	20.0	20.4	102	75 - 130
Chloromethane	20.0	21.3	107	75 - 130
Vinyl Chloride	20.0	22.2	111	75 - 130
Bromomethane	20.0	22.3	112	75 - 130
Chloroethane	20.0	22.0	110	75 - 130
Trichlorofluoromethane	20.0	22.0	110	75 - 130
1,1-Dichloroethene	20.0	22.6	113	75 - 130
Methylene Chloride	20.0	21.5	107	75 - 130
Trans-1,2-Dichloroethene	20.0	21.4	107	75 - 130
1,1-Dichloroethane	20.0	21.4	107	75 - 130
Cis-1,2-Dichloroethene	20.0	22.1	110	75 - 130
Chloroform	20.0	21.0	105	75 - 130
1,2-Dichloroethane	20.0	19.9	99.6	75 - 130
1,1,1-Trichloroethane	20.0	19.4	97	75 - 130
Carbon Tetrachloride	20.0	20.2	101	75 - 130
1,2-Dichloropropane	20.0	21.0	105	75 - 130
Trichloroethene	20.0	21.4	107	75 - 130
Bromodichloromethane	20.0	20.0	100	75 - 130
2-Chloroethyl Vinyl Ether	20.0	20.2	101	75 - 130
Cis-1,3-Dichloropropene	20.0	19.7	98	75 - 130
Trans-1,3-Dichloropropene	20.0	19.8	99	75 - 130
1,1,2-Trichloroethane	20.0	20.7	104	75 - 130
Dibromochloromethane	20.0	20.9	105	75 - 130
Tetrachloroethene	20.0	20.7	103	75 - 130
Chlorobenzene	20.0	21.3	107	75 - 130
Bromoform	20.0	22.7	113	75 - 130
1,1,2,2-Tetrachloroethane	20.0	19.9	99.5	75 - 130
1,3-Dichlorobenzene	20.0	20.6	103	75 - 130
1,4-Dichlorobenzene	20.0	20.9	105	75 - 130
1,2-Dichlorobenzene	20.0	21.0	105	75 - 130

Column to be used to flag recovery values

* Values outside QC limits

COMMENTS: _____

APPENDIX N

LABORATORY MATRIX SPIKE/MATRIX DUPLICATE FORM EXAMPLE

FORM 3
SOIL SEMIVOLATILE MATRIX SPIKE / MATRIX SPIKE DUPLICATE RECOVERY

Northeast Analytical Inc.

M.S. SAMPLE NO.: AJ03258 M / AJ03258 K SDG No.: 06040025

NEA Form ID: S:\FORMS\CATB\CLP-3D.XLS
NEA File ID: S:\FORMS\CATB\CLP-3D.XLS

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	#	MS % REC	QC LIMITS REC.
Phenol						26 - 90
2-Chlorophenol						25 - 102
1,4-Dichlorobenzene	50	0.00	27.9		55.8	28 - 104
N-Nitroso-di-n-propylamine	50	0.00	37.4		74.9	41 - 126
1,2,4 - Trichlorobenzene	50	0.00	39.1		78.2	38 - 107
4- Chloro-3-Methylphenol						26 - 103
Acenapthene	50	0.00	44.2		88.5	31 - 137
4-Nitrophenol						11 - 114
2,4-Dinitrotoluene	50	0.00	38.9		77.8	28 - 89
Pentachlorophenol						17 - 109
Pyrene						35 - 142

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC	#	% RPD	#	QC LIMITS RPD	QC LIMITS REC.
Phenol							35	26 - 90
2-Chlorophenol							50	25 - 102
1,4-Dichlorobenzene	50	27.1210	54.2		2.8		27	28 - 104
N-Nitroso-di-n-propylamine	50	34.7650	69.5		7.4		38	41 - 126
1,2,4 - Trichlorobenzene	50	35.1290	70.3		10.7		23	38 - 107
4- Chloro-3-Methylphenol							33	26 - 103
Acenapthene	50	43.0880	86.2		2.62		19	31 - 137
4-Nitrophenol							50	11 - 114
2,4-Dinitrotoluene	50	34.9980	70.0		10.6		47	28 - 89
Pentachlorophenol							47	17 - 109
Pyrene							36	35 - 142

Column to be used to flag recovery values
* Values outside QC limits

Spike Recovery: 0 out of 10 outside limits.
RPD: 0 out of 5 outside limits.

COMMENTS: _____

APPENDIX O

SYSTEM AUDIT EVALUATION REPORT (SAER)

Northeast Analytical, Inc.

REPORT #: _____

Quality Assurance/Quality Control

DATE: _____

System Audit Evaluation

Date of Evaluation: _____ Analytical Method(s): _____

Period Evaluated: _____ to _____ Matrix: _____

System Parameters Evaluated:

[1] Instrument maintenance schedules
[2] Instrument calibration frequency
[3] QC control sample frequency
[4] QC method blank frequency
[5] QC reagent check frequency
[6] QC control chart/control limit update frequency
[7] QC sample container check frequency
[8] Reagent preparation frequency
[9] Standard preparation frequency
[10] Sample Chain of Custody completeness
[11] Glassware/Equipment decontamination procedures
[12] Other: _____

Synopsis of System Audit:

_____

_____

_____

_____

_____

_____

_____

Corrective Action Report Issued?: [Yes] or [No] Ref. # if yes: _____

Review Signatures: Date:

Preparation Chemist(s): _____ _____

Instrument Analyst(s): _____ _____

Section Supervisor: _____ _____

QA Officer: _____ _____

Lab Director: _____ _____

APPENDIX P

PERFORMANCE AUDIT REPORT (PAR)

Northeast Analytical, Inc.

REPORT #: _____

Quality Assurance/Quality Control

DATE: _____

Performance Audit Evaluation

Analytical Method: _____
Sample Matrix: _____
Sample ID Number(s): _____
Date Samples Submitted: _____
Analyst(s): _____

Extraction Logbook Entries Complete?:
(OK for acceptable, NO for not acceptable)

1. Date _____
2. Sample ID _____
3. Sample Mass/Volume _____
4. Extraction Method _____
5. Analytical Method _____
6. Matrix _____
7. QC Standard Addition
   (i) Matrix Spike _____
   (ii) Surrogate _____

Comments: _____
_____
_____

Extraction/Sample Processing Observed?: [Yes] or [No]

Initials of observer: _____
Date: _____

Deviations from Standard Operating Procedure?: [Yes] or [No] (Explain if Yes)

Comments: _____
_____
_____

Analyst/Instrument Logbook entries complete? [Yes] or [No] (Explain if No)

Comments: _____
_____
_____

Final Data Package complete?: [Yes] or [No] (Explain if No)

Comments: _____
_____
_____

Analytical Sequence in order?: [Yes] or [No] (Explain if No)

Comments: _____
_____
_____

Calibration, Check Standards,
Performance Evaluation Standards
And Retention Times in criteria?: [Yes] or [No] (Explain if No)

Comments: _____
_____
_____

Synopsis of Analytical results:

Analyte percent recovery in criteria?: [Yes] or [No]

List outliers below if NO:

ID #	Compound	% Recovery	Limits

Continue on reverse

Matrix Spike percent recovery in criteria?: [Yes] or [No]

List outliers below if NO:

ID #	Compound	% Recovery	Limits

Surrogate percent recovery in criteria?: [Yes] or [No]

List outliers below if NO:

ID #	Surrogate	% Recovery	Limits

Duplicate relative % difference in criteria?: [Yes] or [No]

List outliers below if NO:

ID #	Compound	RPD %	Limits

Surrogate percent recovery in criteria?: [Yes] or [No]
(At least four replicates for RSD)

List outliers below if NO:

ID # (S)	Compound	RSD %	Limits

Comments: _____
_____
_____
_____
_____
_____
_____
_____

Corrective Action Report Issued?: [Yes] or [No] Ref. # if yes: _____

Review Signatures: Date:

Preparation Chemist(s): _____ _____
Instrument Analyst(s): _____ _____
Section Supervisor: _____ _____
QA Officer: _____ _____
Lab Director: _____ _____

APPENDIX Q

NYS ELAP/NELAP CERTIFICATIONS

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2007
Issued April 01, 2006
Revised March 05, 2007

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. ROBERT E. WAGNER
NORTHEAST ANALYTICAL INC
2190 TECHNOLOGY DRIVE
SCHENECTADY, NY 12308

NY Lab Id No: 11078
EPA Lab Code: NY00906

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Amines	
Pyridine	EPA 8270
Benzidines	
3,3' -Dichlorobenzidine	EPA 625
	EPA 8270
Chlorinated Hydrocarbon Pesticides	
4,4'-DDD	EPA 608
	EPA 8081A
4,4'-DDE	EPA 608
	EPA 8081A
4,4'-DDT	EPA 608
	EPA 8081A
Aldrin	EPA 608
	EPA 8081A
alpha-BHC	EPA 608
	EPA 8081A
beta-BHC	EPA 608
	EPA 8081A
Chlordane Total	EPA 608
	EPA 8081A
delta-BHC	EPA 608
	EPA 8081A
Dieldrin	EPA 608

Chlorinated Hydrocarbon Pesticides	
Dieldrin	EPA 8081A
Endosulfan I	EPA 608
	EPA 8081A
Endosulfan II	EPA 608
	EPA 8081A
Endosulfan sulfate	EPA 608
	EPA 8081A
Endrin	EPA 608
	EPA 8081A
Endrin aldehyde	EPA 608
	EPA 8081A
Heptachlor	EPA 608
	EPA 8081A
Heptachlor epoxide	EPA 608
	EPA 8081A
Lindane	EPA 608
	EPA 8081A
Methoxychlor	EPA 608
	EPA 8081A
Toxaphene	EPA 608
	EPA 8081A

Serial No.: 31932

Property of the New York State Department of Health. Valid only at the address shown. Must be conspicuously posted. Valid certificates have a raised seal. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2007
Issued April 01, 2006
Revised March 05, 2007

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. ROBERT E. WAGNER
NORTHEAST ANALYTICAL INC
2190 TECHNOLOGY DRIVE
SCHENECTADY, NY 12308

NY Lab Id No: 11078
EPA Lab Code: NY00906

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Chlorinated Hydrocarbons	
1,2,4-Trichlorobenzene	EPA 625
	EPA 8270
2-Chloronaphthalene	EPA 625
	EPA 8270
Hexachlorobenzene	EPA 625
	EPA 8270
Hexachlorobutadiene	EPA 625
	EPA 8270
Hexachlorocyclopentadiene	EPA 625
	EPA 8270
Hexachloroethane	EPA 625
	EPA 8270
Chlorophenoxy Acid Pesticides	
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
Fuel Oxygenates	
Methyl tert-butyl ether	EPA 8260B
t-Butyl alcohol	EPA 8260B
Haloethers	
4-Bromophenylphenyl ether	EPA 625
	EPA 8270

Haloethers	
4-Chlorophenylphenyl ether	EPA 625
	EPA 8270
Bis (2-chloroisopropyl) ether	EPA 625
	EPA 8270
Bis(2-chloroethoxy)methane	EPA 625
	EPA 8270
Bis(2-chloroethyl)ether	EPA 625
	EPA 8270
Mineral	
Calcium Hardness	EPA 200.7
Hardness, Total	EPA 200.7
Nitroaromatics and Isophorone	
2,4-Dinitrotoluene	EPA 625
	EPA 8270
2,6-Dinitrotoluene	EPA 625
	EPA 8270
Isophorone	EPA 625
	EPA 8270
Nitrobenzene	EPA 625
	EPA 8270

Serial No.: 31932

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2007
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Revised March 05, 2007

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. ROBERT E. WAGNER
NORTHEAST ANALYTICAL INC
2190 TECHNOLOGY DRIVE
SCHENECTADY, NY 12308

NY Lab Id No: 11078
EPA Lab Code: NY00906

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Nitrosoamines	
N-Nitrosodi-n-propylamine	EPA 625
	EPA 8270
N-Nitrosodiphenylamine	EPA 625
	EPA 8270
Phthalate Esters	
Benzyl butyl phthalate	EPA 625
	EPA 8270
Bis(2-ethylhexyl) phthalate	EPA 625
	EPA 8270
Diethyl phthalate	EPA 625
	EPA 8270
Dimethyl phthalate	EPA 625
	EPA 8270
Di-n-butyl phthalate	EPA 625
	EPA 8270
Di-n-octyl phthalate	EPA 625
	EPA 8270
Polychlorinated Biphenyls	
PCB-1016	EPA 608
	EPA 8082
PCB-1221	EPA 608
	EPA 8082
Polychlorinated Biphenyls	
PCB-1232	EPA 608
	EPA 8082
PCB-1242	EPA 608
	EPA 8082
PCB-1248	EPA 608
	EPA 8082
PCB-1254	EPA 608
	EPA 8082
PCB-1260	EPA 608
	EPA 8082
Polynuclear Aromatics	
Acenaphthene	EPA 625
	EPA 8270
Acenaphthylene	EPA 625
	EPA 8270
Anthracene	EPA 625
	EPA 8270
Benzo(a)anthracene	EPA 625
	EPA 8270
Benzo(a)pyrene	EPA 625
	EPA 8270
Benzo(b)fluoranthene	EPA 625

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Polynuclear Aromatics	
Benzo(b)fluoranthene	EPA 8270
Benzo(ghi)perylene	EPA 625
	EPA 8270
Benzo(k)fluoranthene	EPA 625
	EPA 8270
Chrysene	EPA 625
	EPA 8270
Dibenzo(a,h)anthracene	EPA 625
	EPA 8270
Fluoranthene	EPA 625
	EPA 8270
Fluorene	EPA 625
	EPA 8270
Indeno(1,2,3-cd)pyrene	EPA 625
	EPA 8270
Naphthalene	EPA 625
	EPA 8270
Phenanthrene	EPA 625
	EPA 8270
Pyrene	EPA 625
	EPA 8270

Priority Pollutant Phenols	
2,4,5-Trichlorophenol	EPA 625
	EPA 8270
2,4,6-Trichlorophenol	EPA 625
	EPA 8270
2,4-Dichlorophenol	EPA 625
	EPA 8270
2,4-Dimethylphenol	EPA 625
	EPA 8270
2,4-Dinitrophenol	EPA 625
	EPA 8270
2-Chlorophenol	EPA 625
	EPA 8270
2-Methyl-4,6-dinitrophenol	EPA 625
	EPA 8270
2-Nitrophenol	EPA 625
	EPA 8270
4-Chloro-3-methylphenol	EPA 625
	EPA 8270
4-Nitrophenol	EPA 625
	EPA 8270
Cresols, Total	EPA 8270
Pentachlorophenol	EPA 625
	EPA 8270

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Priority Pollutant Phenols	
Phenol	EPA 625
	EPA 8270
Purgeable Aromatics	
1,2-Dichlorobenzene	EPA 602
	EPA 624
	EPA 625
	EPA 8021B
	EPA 8260B
	EPA 8270
1,3-Dichlorobenzene	EPA 602
	EPA 624
	EPA 625
	EPA 8021B
	EPA 8260B
	EPA 8270
1,4-Dichlorobenzene	EPA 602
	EPA 624
	EPA 625
	EPA 8021B
	EPA 8260B
	EPA 8270
Benzene	EPA 602

Purgeable Aromatics	
Benzene	EPA 624
	EPA 8021B
	EPA 8260B
Chlorobenzene	EPA 624
	EPA 8260B
Ethyl benzene	EPA 602
	EPA 624
	EPA 8021B
	EPA 8260B
Toluene	EPA 602
	EPA 624
	EPA 8021B
	EPA 8260B
Total Xylenes	EPA 602
	EPA 624
	EPA 8021B
	EPA 8260B
Purgeable Halocarbons	
1,1,1-Trichloroethane	EPA 624
	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 624
	EPA 8260B

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Purgeable Halocarbons	
1,1,2-Trichloroethane	EPA 624
	EPA 8260B
1,1-Dichloroethane	EPA 624
	EPA 8260B
1,1-Dichloroethene	EPA 624
	EPA 8260B
1,2-Dichloroethane	EPA 624
	EPA 8260B
1,2-Dichloropropane	EPA 624
	EPA 8260B
2-Chloroethylvinyl ether	EPA 624
	EPA 8260B
Bromodichloromethane	EPA 624
	EPA 8260B
Bromoform	EPA 624
	EPA 8260B
Bromomethane	EPA 624
	EPA 8260B
Carbon tetrachloride	EPA 624
	EPA 8260B
Chloroethane	EPA 624
	EPA 8260B
Chloroform	EPA 624

Purgeable Halocarbons	
Chloroform	EPA 8260B
Chloromethane	EPA 624
	EPA 8260B
cis-1,2-Dichloroethene	EPA 624
	EPA 8260B
cis-1,3-Dichloropropene	EPA 624
	EPA 8260B
Dibromochloromethane	EPA 624
	EPA 8260B
Dichlorodifluoromethane	EPA 624
	EPA 8260B
Methylene chloride	EPA 624
	EPA 8260B
Tetrachloroethene	EPA 624
	EPA 8021B
	EPA 8260B
trans-1,2-Dichloroethene	EPA 624
	EPA 8260B
trans-1,3-Dichloropropene	EPA 624
	EPA 8260B
Trichloroethene	EPA 624
	EPA 8021B
	EPA 8260B

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Purgeable Halocarbons	
Trichlorofluoromethane	EPA 624
	EPA 8260B
Vinyl chloride	EPA 624
	EPA 8260B
Purgeable Organics	
2-Butanone (Methylethyl ketone)	EPA 8260B
Residue	
Solids, Total	EPA 160.3
Solids, Total Dissolved	EPA 160.1
Solids, Total Suspended	EPA 160.2
Wastewater Metals I	
Barium, Total	EPA 200.7
	EPA 6010B
Cadmium, Total	EPA 200.7
	EPA 213.2
	EPA 6010B
Calcium, Total	EPA 200.7
	EPA 6010B
Chromium, Total	EPA 200.7
	EPA 6010B
Copper, Total	EPA 200.7

Wastewater Metals I	
Copper, Total	EPA 6010B
Iron, Total	EPA 200.7
	EPA 6010B
Lead, Total	EPA 200.7
	EPA 200.9
	EPA 239.2
	EPA 6010B
	EPA 7421
Magnesium, Total	EPA 200.7
	EPA 6010B
Manganese, Total	EPA 200.7
	EPA 6010B
Nickel, Total	EPA 200.7
	EPA 6010B
Potassium, Total	EPA 200.7
	EPA 6010B
Silver, Total	EPA 200.7
	EPA 6010B
Sodium, Total	EPA 200.7
	EPA 6010B
Strontium, Total	EPA 200.7
	EPA 6010B

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Wastewater Metals II

Aluminum, Total	EPA 200.7
	EPA 6010B
Antimony, Total	EPA 200.7
	EPA 6010B
Arsenic, Total	EPA 200.7
	EPA 200.9
	EPA 6010B
Beryllium, Total	EPA 200.7
Chromium VI	EPA 7196A
	SM 18-19 3500-Cr D
	SM 20 3500-Cr B
Mercury, Total	EPA 245.2
	EPA 7470A
Selenium, Total	EPA 200.7
	EPA 270.2
	EPA 6010B
	EPA 7740
Vanadium, Total	EPA 200.7
	EPA 6010B
Zinc, Total	EPA 200.7
	EPA 6010B

Wastewater Metals III

Cobalt, Total	EPA 200.7
	EPA 6010B
Molybdenum, Total	EPA 200.7
	EPA 6010B
Thallium, Total	EPA 200.7
	EPA 200.9
	EPA 6010B
Tin, Total	EPA 200.7
	EPA 6010B
Titanium, Total	EPA 200.7
	EPA 6010B

Wastewater Miscellaneous

Boron, Total	EPA 200.7
	EPA 6010B
Hydrogen Ion (pH)	EPA 150.1
	EPA 9040B
Oil & Grease Total Recoverable	EPA 1664A
Organic Carbon, Total	EPA 415.1

Sample Preparation Methods

EPA 3010A EPA 3005A EPA 3020A

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All approved analytes are listed below:

Characteristic Testing	
Ignitability	EPA 1010
	EPA 1030
Reactivity	SW-846 Ch7, Sec. 7.3
Chlorinated Hydrocarbon Pesticides	
4,4'-DDD	EPA 8081A
4,4'-DDE	EPA 8081A
4,4'-DDT	EPA 8081A
Aldrin	EPA 8081A
alpha-BHC	EPA 8081A
beta-BHC	EPA 8081A
Chlordane Total	EPA 8081A
delta-BHC	EPA 8081A
Dieldrin	EPA 8081A
Endosulfan I	EPA 8081A
Endosulfan II	EPA 8081A
Endosulfan sulfate	EPA 8081A
Endrin	EPA 8081A
Endrin aldehyde	EPA 8081A
Heptachlor	EPA 8081A
Heptachlor epoxide	EPA 8081A
Lindane	EPA 8081A
Methoxychlor	EPA 8081A
Chlorinated Hydrocarbon Pesticides	
Toxaphene	EPA 8081A
Chlorinated Hydrocarbons	
1,2,4-Trichlorobenzene	EPA 8270
2-Chloronaphthalene	EPA 8270
Hexachlorobenzene	EPA 8270
Hexachlorobutadiene	EPA 8270
Hexachlorocyclopentadiene	EPA 8270
Hexachloroethane	EPA 8270
Haloethers	
4-Bromophenylphenyl ether	EPA 8270
4-Chlorophenylphenyl ether	EPA 8270
Bis (2-chloroisopropyl) ether	EPA 8270
Bis(2-chloroethoxy)methane	EPA 8270
Metals I	
Barium, Total	EPA 6010B
Cadmium, Total	EPA 6010B
Calcium, Total	EPA 6010B
Chromium, Total	EPA 6010B
Copper, Total	EPA 6010B
Iron, Total	EPA 6010B
Lead, Total	EPA 6010B

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Metals I	
Magnesium, Total	EPA 6010B
Manganese, Total	EPA 6010B
Nickel, Total	EPA 6010B
Potassium, Total	EPA 6010B
Silver, Total	EPA 6010B
Sodium, Total	EPA 6010B
Metals II	
Aluminum, Total	EPA 6010B
Antimony, Total	EPA 6010B
Arsenic, Total	EPA 6010B
Beryllium, Total	EPA 6010B
Chromium VI	EPA 7196A
Mercury, Total	EPA 7471A
Selenium, Total	EPA 6010B
Vanadium, Total	EPA 6010B
Zinc, Total	EPA 6010B
Metals III	
Cobalt, Total	EPA 6010B
Molybdenum, Total	EPA 6010B
Thallium, Total	EPA 6010B
Tin, Total	EPA 6010B
Miscellaneous	
Hydrogen Ion (pH)	EPA 9040B
	EPA 9045C
Lead in Paint	EPA 6010B
Oil & Grease Total Recoverable	EPA 9070
Nitroaromatics and Isophorone	
2,4-Dinitrotoluene	EPA 8270
2,6-Dinitrotoluene	EPA 8270
Isophorone	EPA 8270
Nitrobenzene	EPA 8270
Nitrosoamines	
N-Nitrosodi-n-propylamine	EPA 8270
Phthalate Esters	
Benzyl butyl phthalate	EPA 8270
Bis(2-ethylhexyl) phthalate	EPA 8270
Diethyl phthalate	EPA 8270
Dimethyl phthalate	EPA 8270
Di-n-butyl phthalate	EPA 8270
Di-n-octyl phthalate	EPA 8270
Polychlorinated Biphenyls	
PCB-1016	EPA 8082

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Polychlorinated Biphenyls	
PCB-1221	EPA 8082
PCB-1232	EPA 8082
PCB-1242	EPA 8082
PCB-1248	EPA 8082
PCB-1254	EPA 8082
PCB-1260	EPA 8082
Polynuclear Aromatic Hydrocarbons	
Acenaphthene	EPA 8270
Acenaphthylene	EPA 8270
Anthracene	EPA 8270
Benzo(a)anthracene	EPA 8270
Benzo(a)pyrene	EPA 8270
Benzo(b)fluoranthene	EPA 8270
Benzo(ghi)perylene	EPA 8270
Benzo(k)fluoranthene	EPA 8270
Chrysene	EPA 8270
Dibenzo(a,h)anthracene	EPA 8270
Fluoranthene	EPA 8270
Fluorene	EPA 8270
Indeno(1,2,3-cd)pyrene	EPA 8270
Naphthalene	EPA 8270
Phenanthrene	EPA 8270
Polynuclear Aromatic Hydrocarbons	
Pyrene	EPA 8270
Priority Pollutant Phenols	
2,4,5-Trichlorophenol	EPA 8270
2,4,6-Trichlorophenol	EPA 8270
2,4-Dichlorophenol	EPA 8270
2,4-Dimethylphenol	EPA 8270
2,4-Dinitrophenol	EPA 8270
2-Chlorophenol	EPA 8270
2-Methyl-4,6-dinitrophenol	EPA 8270
2-Methylphenol	EPA 8270
2-Nitrophenol	EPA 8270
4-Chloro-3-methylphenol	EPA 8270
4-Nitrophenol	EPA 8270
Pentachlorophenol	EPA 8270
Phenol	EPA 8270
Purgeable Aromatics	
1,2,4-Trimethylbenzene	EPA 8021B
	EPA 8260B
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1,3,5-Trimethylbenzene	EPA 8021B
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1,3-Dichlorobenzene	EPA 8260B

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Purgeable Aromatics	
1,4-Dichlorobenzene	EPA 8260B
Benzene	EPA 8021B
	EPA 8260B
Chlorobenzene	EPA 8260B
Ethyl benzene	EPA 8021B
	EPA 8260B
Isopropylbenzene	EPA 8021B
	EPA 8260B
n-Butylbenzene	EPA 8021B
	EPA 8260B
n-Propylbenzene	EPA 8021B
	EPA 8260B
p-Isopropyltoluene (P-Cymene)	EPA 8021B
	EPA 8260B
sec-Butylbenzene	EPA 8021B
	EPA 8260B
Styrene	EPA 8260B
tert-Butylbenzene	EPA 8021B
	EPA 8260B
Toluene	EPA 8021B
	EPA 8260B
Total Xylenes	EPA 8021B
	EPA 8260B

Purgeable Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 8260B
1,1,1-Trichloroethane	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 8260B
1,1,2-Trichloroethane	EPA 8260B
1,1-Dichloroethane	EPA 8260B
1,1-Dichloroethene	EPA 8260B
1,2-Dichloroethane	EPA 8260B
1,2-Dichloropropane	EPA 8260B
2-Chloroethylvinyl ether	EPA 8260B
Bromodichloromethane	EPA 8260B
Bromoform	EPA 8260B
Bromomethane	EPA 8260B
Carbon tetrachloride	EPA 8260B
Chloroethane	EPA 8260B
Chloroform	EPA 8260B
Chloromethane	EPA 8260B
cis-1,3-Dichloropropene	EPA 8260B
Dibromochloromethane	EPA 8260B
Dichlorodifluoromethane	EPA 8260B
Methylene chloride	EPA 8260B
Tetrachloroethene	EPA 8260B
trans-1,3-Dichloropropene	EPA 8260B
Trichloroethene	EPA 8260B

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Purgeable Halocarbons	
Trichlorofluoromethane	EPA 8260B
Vinyl chloride	EPA 8260B
Purgeable Organics	
4-Methyl-2-Pentanone	EPA 8260B
Methyl tert-butyl ether	EPA 8021B
	EPA 8260B
Semi-Volatile Organics	
Dibenzofuran	EPA 8270

Sample Preparation Methods

EPA 1310 EPA 1311

Serial No.: 31933

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H.

Expires 12:01 AM April 01, 2007
Issued April 01, 2006
Revised October 23, 2006

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. ROBERT E. WAGNER
NORTHEAST ANALYTICAL INC
2190 TECHNOLOGY DRIVE
SCHENECTADY, NY 12308

NY Lab Id No: 11078
EPA Lab Code: NY00906

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES POTABLE WATER All approved analytes are listed below:

Drinking Water Metals I	
Arsenic, Total	EPA 200.9
Barium, Total	EPA 200.7
Cadmium, Total	EPA 200.7
Chromium, Total	EPA 200.7
Copper, Total	EPA 200.7
Iron, Total	EPA 200.7
Lead, Total	EPA 200.9
Manganese, Total	EPA 200.7
Mercury, Total	EPA 245.2
Selenium, Total	EPA 200.9
Silver, Total	EPA 200.7
Zinc, Total	EPA 200.7
Drinking Water Metals II	
Antimony, Total	EPA 200.9
Nickel, Total	EPA 200.7
Thallium, Total	EPA 200.9
Drinking Water Metals III	
Sodium, Total	EPA 200.7
Drinking Water Non-Metals	
Hydrogen Ion (pH)	EPA 150.1

Volatile Aromatics	
1,2,3-Trichlorobenzene	EPA 502.2
1,2,4-Trichlorobenzene	EPA 502.2
1,2,4-Trimethylbenzene	EPA 502.2
1,2-Dichlorobenzene	EPA 502.2
1,3,5-Trimethylbenzene	EPA 502.2
1,3-Dichlorobenzene	EPA 502.2
1,4-Dichlorobenzene	EPA 502.2
2-Chlorotoluene	EPA 502.2
4-Chlorotoluene	EPA 502.2
Benzene	EPA 502.2
Bromobenzene	EPA 502.2
Chlorobenzene	EPA 502.2
Ethyl benzene	EPA 502.2
Hexachlorobutadiene	EPA 502.2
Isopropylbenzene	EPA 502.2
n-Butylbenzene	EPA 502.2
n-Propylbenzene	EPA 502.2
p-Isopropyltoluene (P-Cymene)	EPA 502.2
sec-Butylbenzene	EPA 502.2
Styrene	EPA 502.2
tert-Butylbenzene	EPA 502.2
Toluene	EPA 502.2
Total Xylenes	EPA 502.2

Serial No.: 31050

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H.

Expires 12:01 AM April 01, 2007
Issued April 01, 2006
Revised October 23, 2006

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

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MR. ROBERT E. WAGNER
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2190 TECHNOLOGY DRIVE
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NY Lab Id No: 11078
EPA Lab Code: NY00906

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Volatile Halocarbons

Tetrachloroethene	EPA 502.2
Trichloroethene	EPA 502.2

Serial No.: 31050

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

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CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. ROBERT E. WAGNER
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2190 TECHNOLOGY DRIVE
SCHENECTADY, NY 12308

NY Lab Id No: 11078
EPA Lab Code: NY00906

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES AIR AND EMISSIONS
All approved analytes are listed below:

Polychlorinated Biphenyls

PCB-1016	EPA TO-10A
PCB-1221	EPA TO-10A
PCB-1232	EPA TO-10A
PCB-1242	EPA TO-10A
PCB-1248	EPA TO-10A
PCB-1254	EPA TO-10A
PCB-1260	EPA TO-10A

Serial No.: 31743

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Appendix R

Quality Assurance Plan Test Method References:

(1.) U.S. EPA SW-846 “Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods”, Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

(2.) Standard Methods for the Examination of Water and Wastewater 18th, 19th and 20th Editions. APHA, AWWA and WPCF. 1992, 1995 and 1998

(3.) 40 CFR 136 (Waste Water Methods) revised July 1, 1998.

(4.) 40 CFR 141 (Drinking Water Methods) revised July 1, 2001.

STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

NE006_09.DOC
REVISION NUMBER: 09

STANDARD OPERATING PROCEDURE FOR THE EXTRACTION AND CLEANUP OF AQUEOUS SAMPLES FOR POLYCHLORINATED BIPHENYL (PCB) ANALYSIS USING THE SEPARATORY FUNNEL EXTRACTION TECHNIQUE (EPA- SW-846 METHOD 3510C) FOR SUBSEQUENT ANALYSIS BY METHOD NEA 608 CAP (CONGENER SPECIFIC GREEN BAY PCB ANALYSIS).

February 27, 2007

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STANDARD OPERATING PROCEDURE

Author: Carrie M. Barss (revision)

Aqueous Extractions Supervisor

Northeast Analytical, Inc.

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Sample Receiving Manager / Technician Analyst

NELAP Annual Review:
Date Last Reviewed: 02/27/07
Reviewed by: Lenore Nelson

QA Reviewed and Approved by:

William A. Kotas
Quality Assurance Officer

1.0 Identification of Test Method

Standard Operating Procedure for the Extraction and Cleanup of Aqueous Samples for Polychlorinated Biphenyls (PCBs) using the Separatory Funnel Extraction Method (USEPA SW-846 Method 3510) for Congener Specific PCB analysis by the Green Bay Mass Balance Method (SOP # NE013.doc)

2.0 Applicable Matrix or Matrices

This method is applicable to surface water, groundwater, wastewater and other aqueous samples.

3.0 Detection Limits

Please see consult determinative method (Congener Specific PCB Analysis by Green Bay Mass Balance Method (SOP # NE013.doc)

4.0 Scope and Application

The following procedure is utilized by Northeast Analytical, Inc. for the extraction, extract concentration, and cleanup of all 209 PCB congeners from water/waste-water samples using the Separatory Funnel extraction method for subsequent analysis by the Green Bay Mass Balance Method, NEA SOP # NE013.

5.0 Summary of the Test Method

5.1 A measured volume of sample (usually 1 liter) is adjusted to appropriate pH, transferred into a separatory funnel, then serially extracted 3 times with Dichloromethane via manual agitation.

5.2 The extract is concentrated, dried, exchanged to Hexane and set to final volume. Default set volume is 5mL. Client and/or project specifications may dictate alternative set volume.

5.3 The sample extract is cleaned up using appropriate cleanup steps (acid wash, florisil, elemental sulfur removal with Mercury) and then presented for GC analysis for PCB Congeners.

6.0 Definitions

Matrix Spike:
An aliquot of sample, fortified with a known quantity of the analytes of interest, used to indicate the accuracy of the method by measuring recovery.

Surrogate Compound Spike:
In chemical composition and chromatography similar to the analytes of interest. Usually not found in environmental samples. These compounds are spiked into all samples, standards, blanks, and matrix spike samples prior to analysis. Percent recoveries are calculated for each surrogate.

QC-Quality Control:
A set of measures for each sample within an analysis methodology to assure that the process is in control.

DI water (De-ionized water):
Water free of any analytes that may interfere with the analytical test compounds.

PSI (Pounds per square inch):
A unit used for measuring pressure.

7.0 Interferences

Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of certain plastics. Phthalate esters respond on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Samples and extracts should not be exposed to plastics such as gloves, tubing, coating on clamps, and pipette bulbs, etc.

8.0 Safety

The extraction chemist should have received in-house safety training and should know the location of first aid equipment and emergency spill / clean-up equipment, before handling any apparatus or equipment. Safety glasses and gloves must be worn when handling glassware and samples. Polychlorinated biphenyls (PCBs) have been tentatively classified as known or suspected carcinogens. The extraction chemist must review the Material Safety Data Sheet (MSDS) for PCBs and all reagents used in the procedure before beginning the extractions. All equipment and solvents should be handled within a lab fume hood.

9.0 Equipment and Supplies

9.1 **Separatory Funnel,** 2 Liter, with Teflon stopcock and Teflon stopper.

9.2 **Graduated cylinder,** 1000mL, 100mL Kimax, Kimble#20026-100

9.3 **Beakers,** Assorted Pyrex:100 mL, 600mL and 1000mL, used for liquid containment and pipette storage.

9.4 **Boston bottles,** 125mL volume with Teflon lined cap.

9.5 **Pipettes**; S/P Disposable Serological Borosilicate Pipettes.
5 mL x 1/10 #P4650-15

Disposable glass Pasteur pipette; Hexane rinsed, 9" Krackeler (or equivalent)

9.6 **pH indicator strips:** pH Range: 0-14.0, EM Science # 9590 (or equivalent)

9.7 **10µL Syringe**, gas-tight, Hamilton #80000
500µL Syringe, gas-tight, Hamilton #81217
1000µL Syringe, gas-tight, Hamilton #81317

9.8 **Drying column,** Pyrex chromatographic column with Pyrex glass wool at bottom and a Teflon stopcock.

9.9 **Glass Wool,** Silanized, Supelco #2-0410, solvent washed as per NE159.sop.

9.10 **Powder Funnel,** Pyrex

9.11 **Stainless Steel Spatula**

9.12 **TurboVap Evaporator concentrator tubes (TurboTubes),** Zymark 250mL, 0.5mL endpoint.

9.13 **TurboVap Evaporator,** Zymark #ZW640-3.

9.14 **Vials:** glass, 8dram and 4dram (with polyseal sealed cap), for sample extracts.

9.15 **Vial Rack**: Plastic rack used to hold vials, during all phases of the extract processing.

9.16 **Nitrogen Micro-blow-down Apparatus**

9.17 **Volumetrics**; 5 mL; Pyrex #5640

9.18 **Centrifuge:** International Equipment Co., Model CL. (or equivalent)

9.19 **Wrist Shaker:** Burrell wrist action shaker, Model 75 and 88. (or equivalent)

9.20 **Auto sampler vials**; Scientific resource Inc., # 99468-A or equivalent

GLASSWARE NOTE:

Prior to use all glassware is to be Hexane rinsed. Following the use all reusable glassware associated with these samples is to be Acetone rinsed 3 times. The glassware is then washed with warm water, thoroughly rinsed with RO-water and dried in a drying oven. This glassware includes all 2L Separatory Funnels, TurboTubes, drying columns, volumetrics, and beakers used for the samples. When storing this glassware in between uses, this glassware is segregated from the remaining laboratory glassware and is not to be used for any other samples. In addition, all disposable glassware used for the samples (including 40mL and 4-dram vials, Pasteur pipettes) should be pre-rinsed two times with Hexane and air dried before use.

10.0 Reagents and Standards

10.1 **5N Sodium Hydroxide;** J.T.Baker # 5761-03

10.2 **1:1 Sulfuric acid:** (H_2SO_4)

Preparation: To a beaker containing 500mL cold DI-water slowly and under constant stirring add 500mL concentrated H2SO4. Allow the mixture to cool after preparation.

10.2 **Acetone,** High Purity Solvent (Burdick/Jackson) #UN1090. (or equivalent)

10.4 **Dichloromethane,** High Purity Solvent (Burdick/Jackson) #UN1593

10.5 **Hexane,** High Purity Solvent (Burdick/Jackson) #UN1208. (or equivalent)

10.6 **Sodium Sulfate,** J.T.Baker #3375-05, Solvent washed as per NE039.sop.

10.7 **1:1 Magnesium Sulfate,** EM Science #MX0075-03/**Sodium Sulfate,** J.T.Baker #3375-05, Solvent washed as per NE091.sop

10.8 **Florisil**; fully activated, J.T. Baker #M368-08, Solvent washed as per NEA094.doc (or equivalent)

10.9 **Concentrated Sulfuric Acid,** Mallinckrodt #2468, #UN1830, Solvent washed as per NE174.doc

10.10 **Mercury**; Triple distilled, Mercury Waste Solutions Inc., Solvent washed as per NE175.sop (or equivalent)

11.0 Sample collection, Preservation, Shipment and Storage

The samples should be collected in unpreserved 1 liter glass bottles with Teflon lined cap. Samples should be delivered to the laboratory on ice and should be chilled to 4± 2 Degrees C. Samples are stored refrigerated at the laboratory at 4 ± 2 degrees C. The sample extraction hold time for this method is five days from the date the samples were collected (not the usual seven days).

12.0 Quality Control

12.1 The extraction chemist should have completed an acceptable demonstration of precision and accuracy before performing the method without supervision. The addition of spiking material to a sample or blank must be witnessed by another extraction chemist and signed in the extraction logbook. All surrogates and matrix spikes must meet acceptable QC limits. An approved instructor must also certify the extraction chemist to perform the procedure.

12.2 A Method Blank sample and Lab Control Spike (LCS) must be prepared per each extraction batch or 1 per 20 site samples whichever is more frequent. A matrix spike/matrix spike duplicate (or lab duplicate) should be prepared for every 20 site samples or as per client specified quality assurance project plan (QAPP). Spike default for LCS, MS, MSD is 0.2mL of A1242 @ 1.0ppm. Client and/or project specifications may dictate and alternate spike amount or Aroclor.

12.3 PCB Surrogate Nonachlorobiphenyl is added to each sample prior to extraction to measure extraction/cleanup efficiency. Default surrogate is: 0.5mL of 5'NCBP @ 0.2ppm. Client and/or project specifications may dictate an alternate surrogate concentration.

13.0 Calibration and Standardization

Please see consult determinative method (Congener Specific PCB Analysis by Green Bay Mass Balance Method (SOP # NE013.doc) for Calibration and Standardization.

14.0 Procedure

14.1 SAMPLE PREPARATION

14.1.1 Throughout the entire process it should be noted that if the extraction chemist encounters any problems or difficulties with any samples or steps involved, all work should **STOP!!!** Any problems should be brought to the attention of the supervisor and documented in the sample extraction logbook.

14.1.2 Before any steps are taken; the extraction chemist should first review the sample job folder and check the sample labels versus the original chain of custody. Any discrepancies should be noted in the sample extraction logbook.

14.1.3 Bring samples to room temperature by letting the samples warm up in the laboratory for a minimum of 30 minutes.

14.1.4 Mark the level of the sample on the outside of the sample container with a felt tip pen. Determine the pH of the sample by removing a small amount of sample with a Pasteur pipette (approximately 0.1 mL) and wet a pH indicator strip. The pH should be between 5 and 9. Adjust the pH with 1:1 Sulfuric Acid if the pH is greater than 9 or 5N Sodium Hydroxide if the pH is lass than 5. Record the original pH in the extraction logbook.

14.1.5 Using 1 Liter clear sample bottles, measure out two, 1 Liter aliquots of DI water to be used as an extraction blank and laboratory control spike.

14.2 SAMPLE EXTRACTION

14.2.1 In a chemical fume hood set up Separatory Funnels with Teflon stopcocks and stoppers for each sample on a funnel holder. Rinse with approximately 20 mL of Acetone, drain and discard the rinse solvent. Then rinse again with approximately 20 mL of Dichloromethane that has been drawn from a beaker (do not use the Dichloromethane Squirt Bottle for this step), drain and discard this rinse also. Allow the Separatory Funnels to dry completely.

14.2.2 Rinse one Boston bottle per sample with Dichloromethane and discard rinse solvent. Let the solvent evaporate by laying the bottles in the fume hood. Label each bottle with appropriate Sample ID, cover with tape to eliminate destroying of ID number.

14.2.3 Add 40mL of Dichloromethane to the Separatory Funnel using a 100 mL graduated cylinder. Place one of the correctly labeled Boston bottles under each Separatory Funnel drain in case of any leaks.

14.2.4 Spike surrogate- and matrix-spike compound solutions directly into the sample containers for any laboratory control spikes, matrix spikes and matrix spike duplicates. Record the surrogate- and matrix-spike concentration, the amount spikes, and the spike solution references code in the extraction logbook.

14.2.5 Invert the sample bottle two to three times to thoroughly mix the sample and spiking solutions. Then carefully transfer the sample into the Separatory Funnel, making sure not to splash or allow sample to run down the outside of the glassware.

14.2.6 Stopper the Separatory Funnel, remove from rack and vent quickly by holding it upside down and opening the stopcock (**aiming it into the hood and away from any other samples**) to release

vapor pressure. Keep a secure hold on the Teflon stopper at all times. Shake and vent until minimal if any vapor pressure is released when the stopcock is opened. Shake the Separatory Funnel vigorously for two minutes venting occasionally.

14.2.7 Return the Separatory Funnel back to the rack and remove the stopper. Allow the solvent and water phases to separate for a minimum of 10 minutes. After 2 or 3 minutes swirl the samples by moving the Separatory Funnel in a circular motion creating a vortex in the water which will sink any Dichloromethane trapped on the surface.

14.2.8 Slowly drain the bottom Dichloromethane layer into the pre-rinsed and correctly labeled 125mL Boston bottle.

14.2.9 Add 30mL of Dichloromethane to the sample bottle using a 100mL graduated cylinder. Cap and shake the bottle to rinse. Transfer this rinse to the corresponding Separatory Funnel by carefully pouring.

14.2.10 Repeat steps 14.2.6 through 14.2.9 once more for a total of three shakes.

14.2.11 Cap the Boston bottles and put into a refrigerator to chill the samples.

14.2.12 Empty the wastewater from the Separatory Funnels into a waste container for proper disposal according to the Chemical Hygiene Plan.

14.2.13 Separatory funnels must be rinsed with Acetone and dried in the hood before being washed with warm water, rinsed with RO water and dried in the drying oven.

14.3 SAMPLE EXTRACT DRYING PROCEDURE

14.3.1 All water extracts must be dried to remove residual water prior to concentration and cleanup. Assemble drying columns using Teflon stopcocks, Teflon stopcock seals, and Viton O-rings. Insert a small plug of glass wool in the bottom of the column.

14.3.2 Using a Pyrex powder funnel, add a small amount of sodium sulfate (approximately 1'' to 2'') to the column.

14.3.3 Rinse the column and sodium sulfate with a full column of Hexane, draining the Hexane into a clean rinse bucket and leaving just enough to cover the sodium sulfate.

14.3.4 Rinse TurboTubes with Hexane, label one per sample and set in holders. Place one directly under each column.

14.3.5 Remove the samples from the refrigerator and using a solvent rinsed stainless steel spatula, slowly add a 1:1 mixture of MgSO4and Na2SO4. Cap the bottle and shake, adding more if necessary until the drying agent is free flowing. Depending on the amount of water in the sample it will take between 1 and 7 scoops of 1:1 MgSO4/Na2SO4 to absorb all the water.

14.3.6 Once the drying agent is free flowing, carefully pour the sample into the column. While the bottle is still inverted over the column rinse the threads with a disposable Pasteur pipette volume of Hexane to complete the transfer of sample.

14.3.7 Add three to four disposable Pasteur pipette volumes of Hexane to the Boston bottle, cap and shake. Pour the solvent into the column and rinse the bottle rim. Repeat the bottle rinse twice more then rinse down the insides of the column with Hexane.

14.3.8 Open the stopcock and allow the sample to drain into the TurboTube. DO NOT let the drying column go dry until the end of 14.3.10.

14.3.9 Stop the column from draining just above the Sodium Sulfate. Rinse insides of the column with three to four Pasteur pipette volumes of Hexane.

14.3.10 Using a graduated cylinder, pour 60mL of Hexane into the column to completely rinse the column into the TurboTube. Let the column drain into the TurboTube for several minutes after the solvent has eluted.

14.3.11 Add 0.1 mL of Toluene to the sample extract (6-8 drops from a Pasteur pipette).

14.4. SOLVENT REDUCTION: TURBOVAP EVAPORATOR SYSTEM

14.4.1 The TurboVap evaporator system is used in place of the Kuderna Danish (KD)-concentrator apparatus. The TurboVap evaporator system is used to reduce the sample volume. The TurboVap uses a heated water bath and positive pressure nitrogen flow / vortex action. The unit maintains a slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract, which allows fractional reduction of the solvents without loss of higher boiling point analytes.

14.4.2 Turn the unit on (switch is located on the backside of the unit) and allow to heat up to the specified temperature for individual solvent use. For this procedure the temperature is 38°C $\pm$ 2°C. This is indicated by the "Heating" display light, located above the temperature control knob on the right side of the unit. The system is at the proper temperature when the "At Temperature" light is lit. This is located above the "Heating" display light. To verify this temperature there is a thermometer located next to the TurboVap, which should be checked periodically to assure the desired temperature is being maintained.

14.4.3 As a precaution the TurboVap system regulators should be checked to assure that there is no residual gas pressure within the system and that the gas pressure regulator is off before placing samples in the apparatus. Residual gas pressure may cause splashing and therefore cause cross contamination of samples. To bleed the system of residual gas pressure place an empty TurboTube into the water bath and close the lid. Make sure that the nitrogen gas pressure regulator is closed. Bleed any residual gas until the regulator gauge reads "0" psi. Remove the empty TurboTube.

14.4.4 Wipe down inside of TurboVap with a Hexane wetted paper towel including top lid and pins. Place TurboTubes containing the sample extracts into the TurboVap and close lid. Slowly open the pressure regulator. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the volume reduction.

14.4.5 The process for solvent (Dichloromethane /Hexane) reduction takes approximately 30-45 minutes. DO NOT leave the unit unattended as extracts may be blown to dryness and loss of PCB as well as surrogate- and matrix-spike may occur. Immediately notify the extraction supervisor if an extract is blown to dryness and note the incident in the extraction logbook.

14.4.6 Concentrate the solvent to approximately 10 mL. Remove from the TurboVap. Add about 90 mL of Hexane to the TurboTube.

14.4.7 Make sure the gas pressure regulator is off.

14.4.8 Repeat step 14.4.4. The process for solvent reduction takes approximately 30 minutes.

14.4.9 Concentrate to approximately 5 mL. Remove the samples from the TurboVap and place in the rack. The remaining solvent will consist almost entirely of Hexane, this process of replacing one solvent with another is called a solvent exchange.

14.5 SAMPLE EXTRACT CLEANUP PROCEDURE

Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection contain co-extracted xenobiotics and other interfering substances, which must be removed before accurate chromatographic analysis can be performed. Not all clean-up procedures need to be performed on every sample and several are sample matrix specific. The experience of the analyst combined with the sampling site history should guide the selection of which clean-up procedures are necessary. The extraction chemist records the sequence and number of repeats of cleanup steps performed in the sample logbook. Sample extract cleanups are performed on set volume extracts. The set volume is 5mL for water/wastewater samples.

14.5.1 Florisil Adsorption (Column)

The Florisil column removes co-extracted polar compounds, and residual water.

14.5.1.1 Prepare a Florisil column by using a 5mL disposable pipette as the column. Insert a small plug of glass wool into the bottom of the pipette and position at the bottom using a 1mL disposable pipette that has been pre-rinsed with Hexane.

14.5.1.2 Fill the column to the 3mL mark on the pipette with fully activated Florisil. Tap the side of the 5mL pipette to settle the Florisil.

14.5.1.3 After the Florisil has settled, rinse the column with approximately 20mL of Hexane, use an 8-dram vial or beaker to collect the Hexane rinse.

14.5.1.4 Label a Hexane pre-rinsed 8-dram vial. While not letting the column go dry, place the 8-dram vial under the column. Quantitatively transfer the sample extract to the column. Rinse the TurboTube with two disposable Pasteur pipette volumes of Hexane, and repeat twice more, adding the rinse to the column. **Note:** The column should not be allowed to go dry but the initial rinses should be eluted in such a way that the sample is not diluted up the column.

14.5.1.5 Rinse the column by allowing the sample to elute to approximately 0.5 mL above the Florisil. Fill the column with Hexane. Allow the column to go dry. The volume should be approximately 20 mL.

14.5.1.6 Concentrate the solvent on the nitrogen micro-blow-down apparatus to a volume of approximately 2 mL. It is important to watch the samples closely so that they do not evaporate completely; otherwise, loss of PCBs may occur.

14.5.1.7 Quantitatively transfer the extract to a Hexane pre-rinsed and labeled 5mL volumetric flask. Use Hexane to rinse the vial and set sample to volume, but do not set to volume until after 14.5.1.8.

14.5.1.8 After transfer, but **prior to setting to volume**, add 4.5µL of internal standard (Octachloronapthalene @ 202ppm in Toluene) to each sample using a 10µL syringe.

14.5.1.9 Set to volume and mix well by inverting the volumetric several times.

14.5.1.10 Transfer sample from the volumetric to a Hexane pre-rinsed and correctly labeled 4-dram vial.

14.5.2 Sulfuric Acid Wash

14.5.2.1 The concentrated sulfuric acid treatment removes hydrocarbons and other organic compounds, which are co-extracted with the PCB residues.

14.5.2.2 Add one Pasteur pipette volume full of solvent washed concentrated Sulfuric Acid to each extract vial, and shake by hand in a chemical fume hood for 30 seconds. Then centrifuge for at least 1 minute on setting #4. Transfer the Hexane (upper) layer to a correctly labeled, Hexane pre-rinsed 4-dram vial.

14.5.2.3 Repeat 14.5.1.2 if the sample extract appears to be heavily loaded (opaque) with colored material. Two to three acid washes may be required.
Note: It is entirely possible that all colored material will not be removed from the extract.

14.5.3 Removal of Sulfur Using Mercury

Note: Mercury is a highly toxic metal, all operations involving Mercury should be performed within a chemical fume hood. Prior to using Mercury, the extraction chemist should become acquainted with proper handling and emergency spill/clean-up procedures associated with this metal and must have reviewed the Material Safety Data Sheet (MSDS).

14.5.3.1 Add 1-3 drops of solvent washed Mercury (NE175.doc) to the sample extracts, and cap. Handshake for 15-30 seconds. If the Mercury changes color or breaks up into tiny balls and will not reform the original ball, change the Mercury. To change the Mercury transfer extract into a new correctly labeled, and Hexane pre-rinsed 4-dram vial and add new Mercury to it. Repeat previous step. Place on wrist action shaker for 30 minutes. The sulfur is converted to Mercuric Sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental Sulfur.

14.5.3.2 Remove the sample extracts from the wrist shaker.

14.5.3.3 The precipitated sulfur can be removed from the extract by performing a sulfuric acid clean up (discussed in sections 14.5.2).

14.6 FINAL EXTRACT PREPARATION

14.6.1 Transfer the extract to a Hexane pre-rinsed and correctly labeled final 4-dram vial. Labeled with the client, the test, the dilution factor, the date and the sample ID number.

14.6.2 Transfer approximately 1.0 mL of extract into a labeled 1.5 mL GC autosampler vial.

14.6.3 Submit the GC vials along with the Project folder containing the GC Queue Lab Sheet and a copy of the extraction logbook page to the GC analyst.

14.7 SAMPLE EXTRACT ARCHIVING

14.7.1 After the sample extracts are analyzed, the analyst will return them to the water lab for archiving.

14.7.2 For each extract label one black screw top GC vial with the NEA ID number.

14.7.3 Fill GC vial to rim with extract, and cap.

14.7.3 Store in a GC vial box in freezer.

15.0 Calculations

Please consult determinative method (Congener Specific PCB Analysis by Green Bay Mass Balance Method (SOP # NE013.doc)) for calculations.

16.0 Method Performance

Please consult determinative method (Congener Specific PCB Analysis by Green Bay Mass Balance Method (SOP # NE013.doc)) for Method Performance details.

17.0 Pollution Prevention

Please see NEA168.SOP for pollution prevention measures

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Please consult determinative method (Congener Specific PCB Analysis by Green Bay Mass Balance Method (SOP # NE013.doc) for Data Assessment and Acceptance Criteria for Quality Control Measures.

19.0 Corrective Action for Out-Of-Control Data

Please consult determinative methods (Congener Specific PCB Analysis by Green Bay Mass Balance Method (SOP # NE013.doc) for Corrective Action for Out-Of-Control Data

20.0 Contingencies for Handling Out-Of-Control or Unacceptable Data

Please consult determinative methods (Congener Specific PCB Analysis by Green Bay Mass Balance Method (SOP # NE013.doc) for Contingencies for Handling Out-Of-Control or Unacceptable Data

21.0 Waste Management

Please see NEA054.SOP, NEA083.SOP and NEA089.SOP

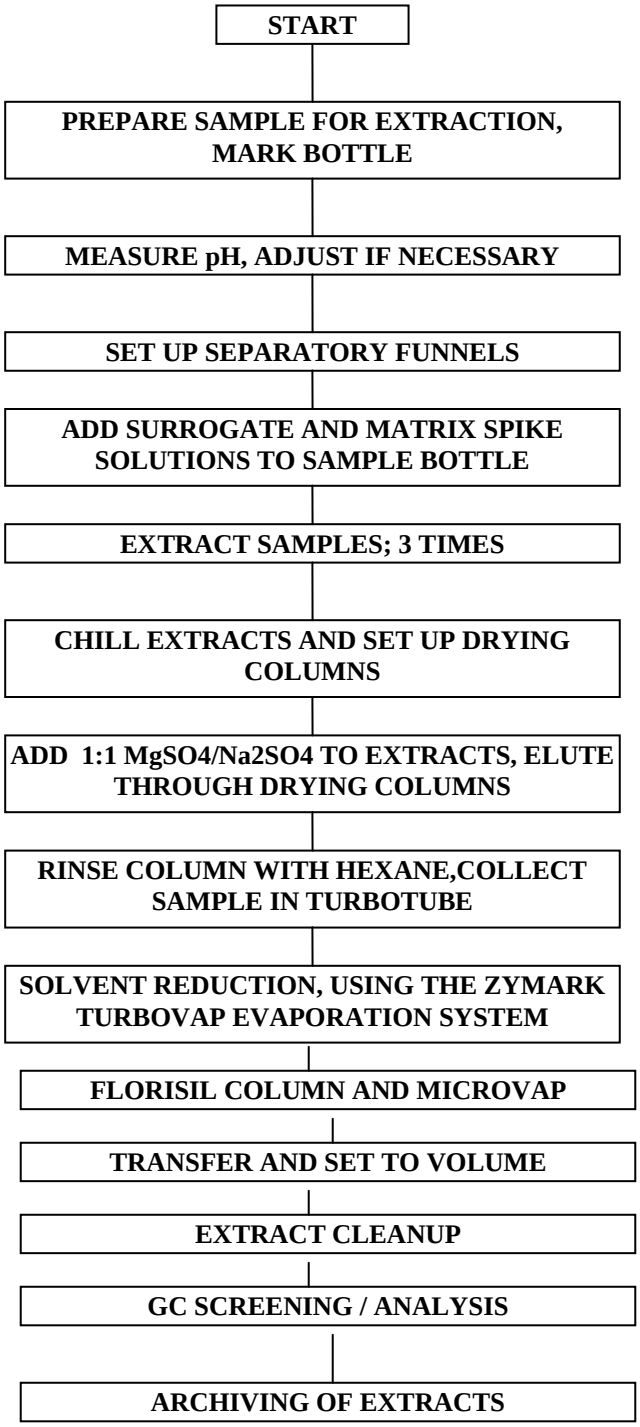
22.0 References

1. U.S. EPA SW-846 “Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods”, Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

2. “Guide to Environmental Analytical Methods”, Fourth Edition, Genium Publishing Corporation, 1998.

23.0 Tables, Diagrams, Flowcharts and Validation Data

23.1 Method Flowchart



STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

NE0017_07.DOC
REVISION NUMBER: 07

Standard Operating Procedures for Extraction and Extract Cleanup of Fish and Biota Materials by EPA 3540 (Soxhlet Extraction) for Polychlorinated Biphenyl (PCB) Analysis

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STANDARD OPERATING PROCEDURE

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Northeast Analytical, Inc.
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Reviewed by:

QA Officer

Approved by:

NELAP Annual Review:
Date last reviewed:
Reviewed by:

William A. Kotas
Quality Assurance Officer

1.0 TITLE/PURPOSE ***Title:***

Standard Operating Procedure for the extraction and cleanup of polychlorinated biphenyls from fish and biota material by SW-846 Method 3540 (Soxhlet Extraction).

Purpose:

This SOP provides the method for the extraction of polychlorinated biphenyls from fish tissue for analysis by EPA Method 8082.

2.0 APPLICABLE MATRICES

This test method is appropriate for all fish and biota material. The chemist must have an understanding of the methods and requirements of USEPA-SW- 846A "Test Methods for Solid Wastes" Volume 1B: Lab Manual, 3rd edition. Methods 3540, 3500, 2500A. An approved instructor must also certify the chemist to perform the procedure. The chemist should have completed an acceptable demonstration of precision and accuracy before performing this method without supervision. This method may be restricted to use by or under the supervision of an extraction chemist knowledgeable in the area of extraction and clean-up. The chemist should further be aware of the proper care and handling of PCBs as well.

3.0 DETECTION LIMIT

Please see determinative method (Lab SOP NE148) for details

4.0 SCOPE AND APPLICATION

The following procedure is used by NEA for the extraction of fish tissue for PCB analysis, although it may be adapted for other biota extractions.

5.0 SUMMARY OF TEST METHOD

The extraction and cleanup of polychlorinated biphenyls from fish and biota material starts with sample prep such as sexing, de-scaling, filleting, and grinding fish or the grinding of any biota material to be tested. Once ready the sample material needs to be weighed and then dried using 1:1 magnesium sulfate and sodium sulfate. The material is then transferred to a pre rinsed cellulose extraction thimble which is then placed inside a soxhlet extractor apparatus to be extracted for 16 hours. Once the extraction is complete the solvent is then exchanged to pure hexane utilizing a LV evaporation system. After solvent exchange, the extract is set to volume and sent through several cleaning procedures including acid, florisil, TBA and mercury. Once clean the solvent extract is set to proper dilutions for GC analysis.

6.0 DEFINITIONS

Blank: A non site sample created in the laboratory to evaluate cross contamination of the sample set

Lab Control Standard: A non site sample created in the laboratory to which a known amount of target analyte is added for assessment of analyte recovery efficiency

Lab Control Standard Duplicate: An exact copy of the Lab Control Standard to further assess analyte recovery efficiency

Matrix Spike: A site sample to which a known amount of target analyte is added for assessment of analyte recovery efficiency.

Matrix Spike Duplicate: An exact copy of the Matrix Spike utilizing the same site sample and known amount of target analyte for assessment of analyte recovery efficiency

7.0 INTERFERENCES Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of certain plastics. Phthalate esters respond on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Samples and extracts should not be exposed to plastics such as gloves, tubing, coating on clamps, and pipette bulbs, etc.

8.0 SAFETY Polychlorinated biphenyls should be treated with extreme caution; as a class of chemical compounds they possess both toxic and suspected carcinogenic properties. The chemist should have received in-house safety training and should know the location of first aid equipment and the emergency spill/clean-up equipment, before handling any apparatus or equipment. Safety glasses and gloves must be worn when handling glassware and samples.

9.0 EQUIPMENT AND SUPPLIES

9.1	Water Cooled Condenser: Pyrex 45/50 #3840-MCO (or equivalent)
9.2	250mL Round Bottom Flask: Pyrex #4100 (or equivalent)
9.3	Soxhlet Repetitive Flushing (reflux) Unit: 45/50 Pyrex #3740-M (or equivalent)
9.4	Heating Mantle: Type "VF" laboratory heating mantle #HM0250VF1 (or equivalent)
9.5	Heating Mantle Controller: Glass-Col #PL3122 Minitwin (or equivalent) regulates temperature control of the mantle.
9.6	Analytical Balance: Mettler PL-303 (or equivalent) used to determine sample mass.
8.7	Cellulose Extraction Thimble: Contains sample during soxhlet extraction.
9.8	Chiller: Pump driven water circulating cooling system cool flow #75 NESLABS Instruments, Inc. (or equivalent)
9.9	TurboVap Evaporator: Zymark #ZW640-3.
9.10	TurboVap Evaporator concentrator tubes: Zymark 250mL, 0.5mL endpoint.
9.11	Beakers: Assorted Pyrex: 250mL, 600mL, and 1000mL, used for liquid containment and pipette storage.
9.12	Vials: glass, 40mL & 4 dram (with polyseal sealed cap) (20 mL & 10 mL) capacity, for sample extracts.
9.13	Vial Rack: Plastic rack used to hold vials, during all phases of the extract processing.
9.14	Centrifuge: International Equipment Co., Model CL. (or equivalent)
9.15	Wrist Shaker: Burrell wrist action shaker, Model 75 and 88. (or equivalent)
9.16	Pipettes: S/P Disposable Serological Borosilicate Pipettes. 1. 1mL X 1/10 2. 5mL X 1/10 3. 10mL X 1/10 Fisher Pasteur Borosilicate glass pipette 9" #72050 (or equivalent)
9.17	Beakers: Assorted Pyrex: 250mL, 600mL, and 1000mL
9.18	4oz. Jars: Industrial Glassware
9.19	Microblowdown Apparatus

10.0 REAGENTS AND STANDARDS

10.1	Sodium Sulfate: J.T.Baker, #3375-05 Anhydrous, Granular (12-60 Mesh) (or equivalent). Used for the laboratory method blank and laboratory control spike.
10.2	Boiling Chips: Chemware PTFE Boiling Stones, P#0919120 (or equivalent)
10.3	Hexane: High Purity Solvent Baxter (Burdick/Jackson) #UN1208. (or equivalent)
10.4	Acetone: High Purity Solvent Baxter (Burdick/Jackson) #UN1090. (or equivalent)
10.5	1:1 Hexane/Acetone: 50%/50% by volume solvent mixture prepared in the lab.
10.6	Florisil: deactivated, Tested and Approved, see NE095.SOP
10.7	TBA Reagent: Tetrabutylammonium Hydrogen-Sulfite Reagent (prepared in the laboratory)
10.8	Mercury: Triple distilled Mercury Waste Solutions, Inc. (or equivalent)
10.9	Sulfuric Acid: Na_2SO_4 (concentrated) Malinkrodt #2468 #UN1830. (or equivalent)
10.10	1:1 Magnesium Sulfate/Sodium Sulfate: 50%/50% by volume solvent mixture prepared in the lab.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT and STORAGE

All samples should remain frozen at all times unless being tested. Fish usually arrive whole bodied or already filleted. Once received the sample must be ground and homogenized so that it may be analyzed. The homogenized fish tissue should be held for 6 to 12 months. The fish solvent extracts should be held for 3 months. Some clients may request that the body and/or head of fish be saved once the fillets are cut out. Other biota material may have other specifications stated specifically for that project.

12.0 QUALITY CONTROL

Each sample set is run with a Blank sample and a Lab Control Standard. Every 20th sample set is run with a Matrix Spike and Matrix Spike Duplicate. Clients also have the option of requesting that a Matrix Spike and Lab Control Standard Duplicate be run with their specific sample set (see section 6.0 Definitions for explanation on what these are).

Please see determinative method (Lab SOP NE148) for details

13.0 CALIBRATION AND STANDARDIZATION

The analytical balance should be calibrated daily to ensure accurate measurements are made when weighing out soil samples for extraction.

Please see determinative method (Lab SOP NE148) for details

14.0 PROCEDURES

14.1 Sample Preparation

14.1.1 Throughout the entire process it should be noted that if the chemist encounters any problems or difficulties with any samples or steps involved, all work should STOP! Any problems should be brought to the attention of the supervisor and documented in the extraction logbook.

14.1.2 Before any steps are taken, the chemist should first review the sample job folder and review the sample container labels versus the client chain of custody.

14.1.3 The fish samples are usually received as fillets and must be processed to produce a homogenous material prior to extraction. Once the sample has been logged in, the sample is processed according to SOP NE132.SOP.
The sample is then placed in the freezer for storage until the extraction process is begun.

14.2 Procedure: Sample Extraction

14.2.1 Rinse the thimbles with Hexane, allow them to dry out in a 4oz. jar in a fume hood.

14.2.2 Fill a Pyrex pan with ice cubes and cold water about 1/2 inch deep. As the samples are weighed out, place the beakers in the Pyrex pan to chill prior to the drying step.

14.2.3 Into a pre-cleaned and tared 250mL beaker or 4oz. jar accurately weigh to the nearest 0.001g, using an analytical balance, between 9-10 grams of tissue with a metal spatula. Record this weight in the laboratory extraction log book. Place the beaker containing the sample into the Pyrex pan to chill. Repeat for remaining samples.
Note: All samples containers are to be returned to the appropriate refrigerator. For all empty containers, see the chemical hygiene plan for proper disposal.

14.2.4 After the sample has been sufficiently chilled, add approximately 10 g. of a 1:1 mix of magnesium sulfate/sodium sulfate to the sample and mix well with the metal spatula. If the sample has not dried after a few minutes, another 10 g. may be added. Once the sample is well-dried and free flowing, transfer the sample to an extraction thimble.
Note: Be careful not to add too much drying agent to the sample, if too much is added, the sample may not fit completely in the thimble. In this case the sample will have to be split into two separate soxhlets.

14.2.5 Add 200 mL of a 1:1 mixture of Hexane/Acetone to a 250 mL round bottom flask. Add several boiling chips. Place a soxhlet extractor on top of the round bottom flask.

Label the round bottom with a sample number and place the corresponding thimble into the soxhlet extractor. After all samples have been processed add the specified surrogate and matrix spikes required directly into thimble.

14.2.6 Rinse the inside and the outside connecting joints of the condenser units that will be used to condense the extraction solvent during the soxhlet extraction of the sample with hexane. Turn on chiller to cool the condensers.

14.2.7 Place the round bottom flask with attached soxhlet extractor onto a heating mantle and attach condenser unit. Turn corresponding thermostats on to setting 5. Double check soxhlets at this time for any cracks or chips, which may leak solvent. Once the solvent begins to boil, a flushing action of once every two to three minutes should be achieved.

14.2.8 The samples should be extracted overnight for a minimum of 16 hours. Once the sample has finished extracting (usually in the morning), turn the heating mantle off and allow samples to cool to room temperature. Once cool, rinse the inside of the condensor with several pipette volumes of Hexane. Disengage the soxhlet and condenser unit and rinse the joint off as well into the soxhlet.

14.2.9 Move soxhlet units into a chemical fume hood and flush the remaining solvent from the soxhlet extractor by tipping the soxhlet. Using a pair of long-handled tweezers, pull the thimbles out of the soxhlets one at a time and allow them to drip dry by balancing the thimbles on the tops of the soxhlets. Once dry, remove the thimbles to a sheet of aluminum foil for total solvent evaporation then dispose of into solid PCB waste.

14.2.10 Rinse the soxhlet with several pipette full of Hexane and tip again to drain into the round bottom. Set aside the soxhlet at this time. Procure the same number of TurboTubes as there are samples. Using an individual TurboTube stand, label a TurboTube with the corresponding sample ID number and place in the holder. To each round bottom add ~10 to 15 grams of sodium sulfate and swirl to get out any excess water. Pour the contents of the round bottom into the Turbo Tube, using a pipette and Hexane to rinse the last drops out of the mouth of the round bottom making sure not to transfer any boiling chips or sodium sulfate. Rinse the round bottom with several pipettefull of Hexane, swirl gently, and decant into same TurboTube. Repeat this step twice for same sample, then repeat all preceding steps for all other samples.

14.2.11 All glassware must be rinsed with technical grade (tech)-Acetone or a "for rinsing-only" labeled solvent, and dried in the hood before other cleaning steps.

14.3 Solvent Reduction: TurboVap Evaporator System

14.3.1 The TurboVap evaporator system is used in place of the Kuderna Danish (KD)-concentrator apparatus. The TurboVap uses a heated water bath and positive pressure nitrogen flow/vortex action. The unit maintains a slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract, which allows fractional

reduction of the solvents without loss of higher boiling point analytes.

14.3.2 Turn the unit on (switch is located on the back side of the unit) and allow to heat up to the specified temperature for individual solvent use. This is indicated by the "Heating" display light, located above the temperate control knob on the right side of the unit. The system is at the proper temperature when the "At Temperature" light is lit. This is located above the "Heating" display light.

14.3.3 As a precaution the TurboVap system regulators should be checked to assure that no residual gas pressure remains within the system and that the gas cylinder valve and gas pressure regulators are both off before placing samples in the apparatus. Residual gas pressure may cause splashing and cross contamination of samples. To bleed the system of residual gas pressure place an empty TurboTube into the water bath and close the lid. Make sure that the nitrogen gas cylinder valve is turned off and slowly turn on the gas pressure regulator. Bleed any residual gas until the regulator output pressure gauge reads "0" psi. Proceed to 8.3.4. Rinse the turbo vap with hexane.

14.3.4 Place the TurboTube containing the samples into the TurboVap and close the lid. Turn on the gas cylinder valve first and then begin slowly turning the pressure regulator on. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the reduction.

14.3.5 The process for solvent (Hexane/Acetone) reduction takes approximately 20-30 minutes. Do not leave the unit unattended as extracts may be blown to dryness and PCB loss may occur. Immediately notify a supervisor if an extract is blown to dryness.

14.3.6 Concentrate the solvent to approximately 10 mL. Remove the samples from the TurboVap and place in the rack. The remaining solvent will consist largely of Hexane since the Acetone component is fractionally removed at a faster rate than Hexane; however, a solvent exchange with Hexane should be completed to ensure the Acetone has been entirely removed. Fill the turbo tube back up with about approximately 200mls of hexane. Proceed to concentrate the solvent to 10mls. Then remove the turbo tube and place in a rack. NOTE: Not all samples will evaporate at the same rate; sample extracts containing large amounts of petroleum or other non-volatile liquids may stop reducing before the 10.0 mL point is achieved. Samples, which stop reducing, should be removed as soon as possible.

14.3.7 Quantitatively transfer the sample extract with a disposable transfer pipette into an appropriate volumetric flask (25mL for biota extracts) with three 2mL Hexane rinses. After the sample has been transferred, rinse the disposable transfer pipette with 0.5mL of Hexane into the volumetric flask. Add Hexane to the volumetric meniscus mark. Invert the volumetric flask at least three times to mix completely. Decant the contents into a pre-labeled 40mL vial.

14.3.8 Once the extracted solvent is set to volume see section 15.0 Calculations for instructions on how to calculate the % lipids for the samples.

14.3.9 All dirty glassware must be rinsed with tech-Acetone or a "For Rinsing-Only" labeled

solvent and dried in the fume hood before being washed.

14.4 Sample Extract Cleanup

Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection contain co-extracted xenobiotics and other interfering substances which must be removed before accurate chromatographic analysis can be performed.
Not all clean-up procedures need to be performed on every sample and several are sample matrix specific. The experience of the analyst combined with the sampling site history should guide the selection of which clean-up procedures are necessary. The sample preparation chemist in the extraction logbook records the sequence and number of repeats of cleanup steps performed.
Sample extract cleanups are performed on set volume extracts. The set volume is 25 mL for biota samples.

14.4.1 Sulfuric Acid Wash

14.4.1.1 The concentrated sulfuric acid treatment removes hydrocarbons and other organic compounds, which are co-extracted with the PCB residues.

14.4.1.2 Chill the sample in the freezer for 10-20 mins. Add 5.0 mL concentrated H_2SO_4 and shake for 30 seconds by hand, centrifuge for approximately 1 minute at a setting of 4, transfer 20mL of the Hexane layer(upper layer) to an 40mL vial. If multiple layers occur, sonic for 15-20mins to help reduce the layering.

14.4.1.3 Repeat 8.4.2 if the sample extract appears to be heavily loaded (opaque) with colored material. Two to three acid washes may be required. Note: it is entirely possible that all colored material will not be removed from the extract.

14.4.2 Elemental Sulfur Clean-up

14.4.2.1 Elemental sulfur is soluble in the extract solvents used for biota samples. It is commonly found in many sediment/soil samples, decaying organic material, and some industrial wastes. Large amounts of sulfur can cause the electron capture detector (ECD) to signal saturate for long periods during the elution envelope of PCBs. Even small amounts of sulfur can interfere with PCB measurement as a co-eluting chromatographic peak.

14.4.2.2 Two techniques exist for the elimination of elemental sulfur in PCB extracts. Mercuric precipitation (Mercury Shake) and the Tetrabutylammonium (TBA) sulfite procedure. Tetrabutylammonium sulfite causes the least amount of degradation of a broad range of pesticides and organics compounds, while mercury may degrade organophosphorus and some organochlorine pesticides. The TBA procedure also has a higher capacity for samples containing high concentrations of elemental sulfur.

14.4.3 Removal of Sulfur Using Mercury

Note: Mercury is a highly toxic metal. All operations involving mercury should be performed within a hood. Prior to using mercury, the chemist should become acquainted with proper handling and emergency spill/clean-up procedures associated with this metal and must have reviewed the material safety data sheet MSDS.

14.4.3.1 Add 1-3 drops of mercury to the sample extracts, cap, and place on the wrist shaker for 30 min. The sulfur is converted to mercuric sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental sulfur.

14.4.3.2 Remove the sample extracts from the wrist shaker and place in the centrifuge for 2 minutes on speed setting on #4.

14.4.3.3 Transfer the sample extract to a clean 40mL vial.

14.4.3.4 The precipitated sulfur can be removed from the extract by performing a sulfuric acid clean-up or Florisil slurry (discussed in 8.8.0).

14.4.4 Removal of Sulfur using TBA Sulfite

14.4.4.1 The TBA procedure removes elemental sulfur by conversion to the thiosulfate ion, which is water soluble.

14.4.4.2 Add 2.0mL TBA Sulfite Reagent, 1.0 mL 2-propanol, and approximately 3 aliquots (using the dedicated dispenser) of sodium sulfite crystals to the extract and shake for at least 5 minutes on the wrist shaker and observe. An excess of sodium sulfite must remain in the sample extract during the procedure. If the sodium sulfite crystals are entirely consumed add one or two more aliquots to the extract and observe.

14.4.4.3 Place the samples on the wrist shaker for 45 minutes observing at 15 minute intervals to make sure that the sodium sulfite is not consumed. Add 5mL organic free water and shake for ten minutes.

14.4.4.4 Place the samples into the centrifuge and spin for approximately 2 minutes on setting #4.

14.4.4.5 Transfer the Hexane layer to a new 40mL vial and cap.

14.4.5 Florisil Adsorption (Slurry)

14.4.5.1 The Florisil slurry removes co-extracted polar compounds, residual water, and residual acid and is recommended as the final cleanup step before the extract is submitted for GC analysis.

14.4.5.2 Add approximately 1.5 grams of tested and approved deactivated Florisil to each vial

containing the sample extract.
SEE SUPERVISOR FOR THE APPROPRIATE FLORISIL DEACTIVATION CONCENTRATION TO BE USED.

14.4.5.3 Vigorously shake the vial for approximately 1 min by hand or on the wrist shaker.

14.4.5.4 Place the vial(s) into the centrifuge for 2 minutes on setting#4.

14.4.5.5 Transfer the extract to a clean 40mL vial.

14.5 Extract Screening and Dilution:

14.5.1 PCB extracts are generally screened by GC initially to determine the approximate concentration before final analysis. Prior site history and client supplied estimates of sample concentration may be used to determine what, if any, extract dilution is necessary. Extracts of unknown concentration are generally screened at a 10 to 100 fold dilution.

14.5.2 The supervising chemist is responsible for determining initial screening dilutions. Extract dilutions are prepared by transferring an aliquot of the original sample extract into a vial containing the correct amount of "make up" volume of Hexane. For example, adding 1.0 mL of the extract to 9.0 mL Hexaneperforms a 1 to 10 dilution. The vial containing the diluted extract is labeled denoting the equivalent extract volume after the dilution; e.g. a 25mL extract diluted 1 to 10 is labeled "250X", an undiluted 25mL extract is labeled "25X".
When high dilutions are prepared, secondary (serial) dilutions of the initial diluent are prepared; e.g. a 100 fold dilution is prepared by a 1 to 10 dilution of the initial extract, then a 1 to 10 dilution of the resulting diluent.

14.5.3 Perform the dilution using an appropriate disposable volumetric pipette to transfer the extract and the make-up volume of Hexane. Make sure that the vial is properly labeled. Cap and invert the vial at least three times to thoroughly mix the extract with the solvent.

14.5.4 Transfer 1 mL of the extract to a labeled 1.5 mL GC autosampler vial. Record the screening dilution on the GC Queue Lab Sheet (Lotus-123 spreadsheet) along with the extract volume, and the sample mass. Submit the GC Queue Lab Sheet with the sample extracts to the GC analyst.

15.0 CALCULATIONS

All fish samples require a % lipid analysis in which 10 ml of the 25ml extraction is used. An initial weight of the empty weight dish must be entered into the LIMS system. Then 10ml of the 25ml fish extract (prior to cleaning steps) is placed into the dish and blown dry using a micro blowdown apparatus. Once blown dry the dish should be placed in a dessicator for a minimum of 4 hours.

When completely dry the final weight must be taken and entered into LIMS in which the lipid content is calculated utilizing the below equation:

$$\% \text{ Lipids} = \frac{(\text{Final Wight} - \text{Initial Wight})}{\text{Sample Wight}} \times \frac{\text{Extracted Volume(10 ml)}}{\text{Whole Volume (25ml)}} \times 100$$

Please see determinative method (Lab SOP NE148) for details

16.0 METHOD PERFORMANCE

Please see determinative method (Lab SOP NE148) for details

17.0 POLLUTION PREVENTION

See NEA.168.SOP

18.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

Please see determinative method (Lab SOP NE148) for details

19.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

Please see determinative method (Lab SOP NE148) for details

20.0 CONTINGINCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Please see determinative method (Lab SOP NE148) for details

21.0 WASTE MANAGEMENT

See NEA054.SOP, NEA083.SOP, and NEA089.SOP

22.0 REFERENCES

1. SW-846 methods 3500A & 3600A; United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Vol.1B, Cincinnati, OH 45268

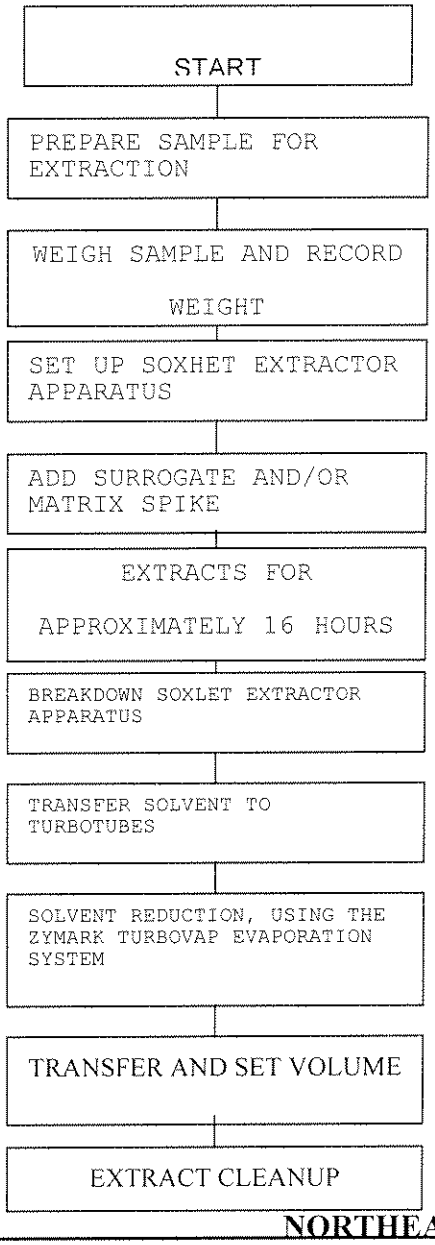
2. Guide to Environmental Analytical Methods, Genium Publishing

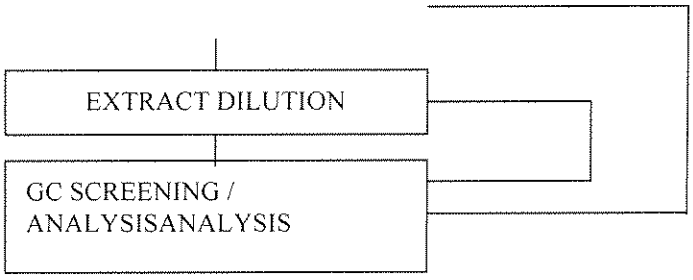
Corporation, Schenectady, NY 12304.

23.0 TABLES, DIAGRAMS, FLOWCHARTS and VALIDATION DATA

1. Method flow chart
2. Note page for analyst

PCBs IN FISH TISSUE/BIOTA
SOXHLET EXTRACTION/TURBO VAP EVAPORATOR




EXTRACT DILUTION
GC SCREENING /
ANALYSISANALYSIS

STANDARD OPERATING PROCEDURE

Author: John Nicpon

Northeast Analytical, Inc.
Issuing section: Inorganics laboratory
NE117_03.SOP
Date: January 6, 2004
Revision Number: 3

Reviewed by:

William A Kotas -Quality Assurance Officer

Approved By:

Robert E. Wagner-Laboratory Director

1.0 TITLE

Standard operating procedure for the determination of Non-Filterable Residue (a.k.a. Total Suspended Solids) according to EPA 1979 method 160.2.

2.0 PURPOSE

The purpose of this SOP is to provide procedures analyzing water samples for suspended solids.

3.0 SCOPE

This method is applicable to waste water and ground water.

4.0 COMMENT

Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103 - 105 ^{o} C. A measured volume of sample is passed through a pre-weighed crucible that contains a glass fiber filter. The crucible is dried in an oven and the change in weight is determined. The concentration of non filterable solids is calculated by dividing the difference in the crucible weight by the volume of sample filtered.

The holding time for analyzing samples is seven days from the date that the sample was collected. The detection limit for samples is dependent on the amount of sample filtered. If a client requires the lowest detection limit possible, the entire sample bottle is to be used.
The Practical Quantitation Limit for a one liter sample is 1.0 mg/L.

Because the method is based on gravimetric measurements, handle the crucibles with tweezers or with vinyl gloves.

5.0 SAFETY

All safety precautions associated with the preparation and analysis of samples must be followed. Gloves and protective eyewear is required.

6.0 REQUIREMENTS

6.1 Method detection limit study

6.1.1 Analyze eight samples (use ERA control samples or other vendor) and a blank according to the procedures set forth in this document. Calculate the MDL by multiplying the standard deviation of eight MDL measurements by 2.998. For the MDL to be valid, it must be greater than 1/10 the amount spiked but not greater than the amount spiked.

6.2 Precision and accuracy studies

6.2.1. Each analyst must demonstrate the ability to generate acceptable results with this method using the following procedure.

6.2.2 Spike four precision and accuracy samples for the analytes of interest at a level at least ten times the PQL level.

6.3 Knowledge on the operation and calibration of the analytical balance is required.

6.4 Knowledge on the operation of the drying oven located in the Inorganics laboratory.

6.5 Knowledge on the maintenance of the portable desiccator box.

6.6 Knowledge on the operation and maintenance of the vacuum pump.

6.7 Familiarity with the procedure for sample collection, preservation and storage requirements as listed in NE027.SOP.

7.0 EQUIPMENT

7.1 Equipment

7.1.1 Small vacuum pump. Located in the Inorganics laboratory.

7.1.2 Large vacuum pump. Located in the Inorganics laboratory.

7.1.3 One liter Erlenmeyer vacuum flasks with crucible holder and glass insert. (Baxter p/n F4375-1L or equivalent). Located in the Inorganics laboratory.

7.1.4 Glass fiber filters. (Baxter p/n F2835-24 or equivalent). Located in the Inorganics laboratory.

7.1.5 Tweezers. Located in the Inorganics laboratory.

7.1.6 Drying oven. Set at a temperature of 103 -105 ^{o}C. Located in the Inorganics laboratory.

7.1.7 Rinse bottle. Filled with laboratory grade water. Located in the Inorganics laboratory.

7.1.8 500 and 250 ml graduated cylinders. Located in the Inorganics laboratory.

7.1.9 Total suspended solids logbook. Located in the Inorganics laboratory.

7.1.10 Crucibles. (VWR p/n 23835-113 or equivalent)

7.2 Reagents and Standards

7.2.1 Laboratory grade water. Located in the cooler room.

7.2.2 Laboratory control sample. Talc. (Fisher p/n T2-500 or equivalent) Located in the Inorganics chemical shelf. Prepare a 100 mg/L solution for the Laboratory control sample.

8.0 PROCEDURE

8.1 Operation and maintenance of vacuum pumps.

8.1.1 Before using vacuum pumps, check that the minimum volume of pump oil is in the pump oil reservoir. For both pumps, empty the collection flasks frequently to prevent water from entering the pump. For the large

vacuum pump, a water trap is used to prevent water from entering the pump. Empty the water trap when necessary.

8.1.2 To operate the large vacuum pump, connect the collect flasks to the vacuum hoses. Before operating the pump the vacuum hoses should be in the closed position. Place the crucibles on the flasks, turn on the pump, turn the desired vacuum hose to the open position and proceed to filter samples as described in **8.5**.

8.1.3 To operate the small vacuum pump, connect the vacuum hose to the pump to the hose connection on the pump. Place the crucible on the flask, turn the pump on, and proceed to filter the sample as describe in **8.5.**

8.2 Oven operation.

8.2.1 Before placing crucibles in the oven, the temperature of the oven must be between 103 and 105 ^{O}C. Check the calibrated thermometer in the glass bottle inside the oven for the oven temperature. Adjust the temperature of the oven if necessary, by slightly turning the control knob. Wait five minutes, check the temperature, repeat the adjustment process until the temperature is within the required temperature range.

8.3 Operation and maintenance of portable desiccator box.

8.3.1 After the crucibles are removed from the oven, they must be placed in the portable desiccator box before they are weighed. The purpose of the desiccator box is to prevent the crucibles from absorbing moisture while they are cooling.

8.3.2 The desiccant material must be dried in the drying oven when the color of the material is a pale blue. Pour the material into a drying pan and placed in the drying oven for a minimum of one hour. Transfer the material to the portable desiccator box, and wait a minimum of one hour for the material to cool to room temperature before placing crucibles in the desiccator box.

8.4 Total suspended solids logbook and preparation of crucibles.

8.4.1 For each day of analysis, a new page of the logbook must be used. Write the date, analyst initials and the 'TSS' at the top of the page. Set up columns with the labels of 'Crucible #'', 'Initial wt. (g)', 'Final wt. (g)', 'NEA #'', 'Initial vol.(ml)', 'Remain vol.(ml)', 'Volume used(ml)'.

8.4.2 The crucibles must be labeled with identification numbers that allows the analyst to identify the crucibles. Place the desired number of crucibles in a glass drying pan. Place filters into the crucibles with tweezers. The rough side of the filter should be facing up. Using the tweezers, place the crucibles on the collection flasks that are connected to the vacuum pump. Turn the pump on and wet the filter with an aliquot of water from the squirt bottle. **Note:** The filter must cover all the holes of the crucible. Turn off the pump and repeat the process with the next crucible. If a hole appears in the filter, replace the filter and repeat the process. Place the crucibles into the drying pan and place the pan in the drying oven for a minimum of three hour. Prepare extra crucibles with each batch.

8.4.3 Remove the crucibles from the oven and place in the portable desiccator box. Wait one hour, and weigh the crucibles on the analytical balance. Record the crucible number and the initial weight (grams) in the logbook. Using Lims record the initial weight in the appropriate location.

8.5 Sample analysis

8.5.1 Remove the samples from the cooler or refrigerator.

8.5.2 If the entire container of sample is to be filtered, mark the volume level with a marker.

8.5.3 The weight of captured residue on the filter must be greater than 1.0 mg. Generally, a sufficient volume of sample is filtered so that a sufficient amount of solid material covers the filter without clogging the filter. If a client requires the lowest detection limit possible, the entire sample bottle is to be used.

8.5.4 Place the crucible on the collection flask, and record the sample number in the logbook. Turn the pump on and squirt RO water onto the filter. Shake the sample bottle and pour into a 500 ml graduated cylinder. Record the volume (ml), and pour the sample onto the filter.

8.5.5 If the additional sample is required for filtering, repeat **8.5.4**. For samples that require the filtration of the complete sample bottle, pour directly from the bottle into the crucible.

8.5.6 Do not overfill the crucible. If the filter clogs, repeat the analysis with a different crucible and use less sample.

8.5.7 After the sample has passed through the filter, rinse the graduated cylinder with aliquots of RO water and pour onto the filter. Rinse filter with several aliquots of RO water from rinse bottle. Turn off the pump and place the crucible into the drying oven. If the entire sample bottle was used, fill the bottle to the mark with water, pour into a graduated cylinder and record the volume.

8.5.8 The crucibles must be in the drying oven a minimum of one hour. Take the crucibles out of the drying oven and follow **8.4.3** and record the final weight (grams) in the logbook.

8.6 Calculations

8.6.1 To calculate the non filterable residue weight (grams), divide the difference of the initial weight and the final weight of the crucible by the total sample volume (ml), and multiply by 1,000,000. Report the result to two significant figures (mg/L).

8.6.2 To calculate the detection limit for the sample, divide 1000 by the sample volume (ml), and report the result to two significant figures (mg/L).

8.7 Quality control

8.7.1 A sample blank is analyzed on each day of analysis. Filter 1000 ml of RO water through one of the crucibles. The calculated value for the blank must be less than 1.00 mg/L. If the result is unacceptable, repeat **8.5.8** for all the samples associated with the blank. If the result is still unacceptable, repeat **8.4** through **8.6** for all samples associated with the blank.

8.7.2 A laboratory control is analyzed on each day of analysis. From the certified value given, filter enough sample for accurate determination. If the result is outside the recovery limits supplied with the control, repeat **8.5.8** for all the samples associated with the control. If the result is still unacceptable, repeat **8.4** through **8.6** for all samples associated with the control. Record the percent recovery on the logbook page.
% recovery = (calculated value/certified value) X 100. (85%-115%).

8.7.3 A sample duplicate is analyzed on each day of analysis or every ten samples, which ever is more frequent. If additional sample is unavailable for duplicate analysis, analyze the laboratory control a second time. Calculate the relative percent difference (RPD). The limit for RPD is 20 %. If the result is unacceptable, repeat **8.5.8** for all the samples associated with the duplicate. If the result is still unacceptable, repeat the analysis of the samples associated with the duplicate. Record the RPD on the notebook page.
RPD = Abs. {(S1 - S2)/ (S1 + S2)} X 200.

8.7.4 Sample results, NEA numbers and the date of analysis are entered into LIMS.

LIMS RESULT TEMPLATE "TSS" COLUM HEADINGS AND DESCRIPTIONS

Column	Description	Column	Description	Column	Description
INTWGT	Initial wt.: sample	**FINWGT**	Final wt. :sample	**TOTVOL**	Total vol.: sample
TSSI	TSS intermediate cal.	**TSS**	Sample result	**TSS_LIW**	Initial wt.: LCS
B_TSS	Blank result	**D_TSS**	Duplicate sample result	**P_TSS**	Precision calculation

L_TSS	LCS % recovery	**TSS-BFW**	Final wt.: blank	**TSS_BIN**	Blank intermediate cal.
TSS_BV	Total vol.: blank	**TSS_DF**	Final wt.: Duplicate	**TSS_DIN**	Dup. Intermediate cal.
TSS-DV	Total vol. Duplicate	**TSS-LFW**	Final wt.: LCS	**TSS_LIN**	LCS intermediate cal.
A_TSS	True value: LCS	**LITSS**	LCS result	**TSS_DIW**	Initial wt.: Duplicate
TSS_LV	Total vol.: LCS	**TSS_BIW**	Initial wt.: Blank		

8.7.5 Analyst must read, understand, and use the latest version of the laboratory's SOPs that relate to their job responsibilities. The SOPs must be read during their initial training, annually, and when the SOPs are revised.

8.7.6 After the completion of the analysis run, the analyst reviews the logbook(s) and the analytical data for completion.

8.7.7 The supervisor/designee reviews the logbook(s) and data and records their initials and review dates on the appropriate documents.

8.7.8 Each QC measurement is reviewed and compared to the appropriate acceptance criteria.

8.7.9 If the acceptance criteria for QC measures has been exceeded for requested analytes and the data is to be reported, the following procedures must be implemented:
8.7.10.1 The quality assurance officer must be notified.
8.7.10.2 The data must be flagged with the appropriate qualifiers and case narrative.
8.7.10.3 The client must be notified about the data.

9.0 REFERENCES

9.1 "Methods of Chemical Analysis of Water and Wastes'," EPA-60/4-79-020, revised March 1983. method # 160.2

9.2 NYSDOH Environmental Laboratory Approval Program Manual item # 271.

10.0 ATTACHMENTS

10.1 **Attachment A**: Summary of procedure for TSS.

ATTACHMENT A

Summary of procedure for TSS

1. Prepare crucibles and place in oven for a minimum of one hour.

2. Remove crucibles from oven and place in desiccator for one hour, weigh crucibles.

3. If client requires entire sample for analysis, mark bottle, else pour portion of sample into 500 ml graduated cylinder.

4. Record volume, place crucible on flask, turn on pump, and pour sample into crucible.

5. Pour additional sample into crucible until sufficient material has collected on top of filter.

6. Place crucibles in oven for a minimum of one hour, place crucibles in desiccator for one hour.

7. Weigh crucibles and calculate results (mg/L) and detection limits (mg/L) to two significant figures.

8. Q.C.: A blank and control for each day of analysis. A sample duplicate on each day of analysis, or every 10 samples.

9. Q.C. sheets for clients requesting documentation: Control and duplicate calculations.

10. Client certificates for clients requesting documentation: Analyst generates certificates using LIMS.

11. Client folders: A copy of notebook page and data result sheet.

11.0 GLOSSARY

11.1 **Residue, non-filterable:** those solids that which are retained by a glass fiber filter and dried to constant weight at 103 -105 ^{o} C.

11.2 **Laboratory control.:** A standard of known concentration that is independent of the standards used for the quantifying samples. It is analyzed at a frequency of once a day.

12.0 POLLUTION AND WASTE MANAGEMENT

12.1 **The reference for Pollution Prevention is NEA 168.SOP.**

12.2 **The reference for Waste Management are NEA054.SOP, NEA083.SOP and NEA089.SOP.**

STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

NE118_07.DOC
REVISION NUMBER: 07

STANDARD OPERATING PROCEDURE FOR THE EXTRACTION AND CLEANUP OF AQUEOUS SAMPLES FOR POLYCHLORINATED BIPHENYLS USING THE CONTINUOUS LIQUID / LIQUID EXTRACTION METHOD (SW-846 METHOD 3520C) FOR SUBSEQUENT ANALYSIS BY METHODS SW-846 8082 OR EPA 680.

January 8, 2007

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STANDARD OPERATING PROCEDURE

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1.0 Identification of Test Method

Standard Operating Procedure for the Extraction and Cleanup of Aqueous Samples for Polychlorinated Biphenyls (PCBs) using the Continuous Liquid /Liquid Extraction Method (USEPA SW-846 METHOD 3520C) for analysis by EPA Method 8082 or EPA 680.

2.0 Applicable Matrix or Matrices

This method is applicable to surface water, groundwater, wastewater and other aqueous samples. Samples containing a high concentration of particulate, organic product or other interferences may require separatory funnel extraction in lieu of this procedure.

3.0 Detection Limits

Please consult determinative methods (EPA Method 8082 (NE148.doc) and EPA Method 680 (NE040.doc) for detection limits.

4.0 Scope and Application

The following procedure is utilized by Northeast Analytical, Inc. for the extraction and cleanup of all PCB Aroclors and all 209 PCB congeners/10 PCB homologs from aqueous samples using the Continuous Liquid/Liquid Extraction method.

5.0 Summary of the Test Method

5.1 A measured volume of sample (usually 1 liter) is adjusted to appropriate pH and placed into a continuous liquid liquid extractor, and extracted with $MeCl_2$ for 2 hours.

5.2 The extract is concentrated, exchanged to Hexane, and set to final volume. Defult set volume is 10mL. Client and/or project specifications may dictate alternative set volume.

5.3 The sample extract is cleaned up using appropriate cleanup steps (acid wash, florisil, elemental sulfur removal with Mercury or TBA) and then presented for GC/GCMS analysis for PCBs.

6.0 Definitions

Matrix Spike:
An aliquot of sample, fortified with a known quantity of the analytes of interest, used to indicate the accuracy of the method by measuring recovery.

Surrogate Compound Spike:
In chemical composition and chromatography similar to the analytes of interest. Usually not found in environmental samples. These compounds are spiked into all samples, standards, blanks, and matrix spike samples prior to analysis. Percent recoveries are calculated for each surrogate.

QC-Quality Control:
A set of measures for each sample within an analysis methodology to assure that the process is in control.

7.0 Interferences

Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of certain plastics. Phthalate esters respond on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Samples and extracts should not be exposed to plastics such as gloves, tubing, coating on clamps, and pipette bulbs, etc.

8.0 Safety

The extraction chemist should have received in-house safety training and should know the location of first aid equipment and emergency spill / clean-up equipment, before handling any apparatus or equipment. Safety glasses and gloves must be worn when handling glassware and samples. Polychlorinated biphenyls (PCBs) have been tentatively classified as known or suspected carcinogens. The extraction chemist must review the Material Safety Data Sheet (MSDS) for PCBs and all reagents used in the procedure before beginning the extractions. All equipment and solvents should be handled within a lab fume hood.

9.0 Equipment and Supplies

9.1 Accelerated One-Step Extractor/Concentrator*;* Corning # 3915-C consisting of:

a) **Jacketed Concentrator Tube**; Corning # 2157-100TJ
b) **Membrane Assembly**, 47 mL; Corning # 3918-47
c) **Extractor Body**, Corning # 3915-MBO
d) **Extractor Cup**; Corning # 3915-ECO
e) **Snyder Sidearm**; Corning # 3915-MSCO
f) **Coupler Assembly**; Corning # 3915-CAO
g) **Photoceram Membrane Support**; Corning # 3918-47MS
h) **Stopcock Assembly**; Corning # 3915-SCA

i) **Connectors/Washers for Stopcock Assembly**; Corning # 3915-CW
j) **35/25 Joint O-Ring, Viton**; Corning # 3915-35VO
k) **Coupler O-Ring**; Corning # 3915-CVO
l) **35/25 Joint Clamp**; Corning # 2140-35
m) **Hose Connectors for Jacketed Concentrator Tube**; Corning # 158-CO
n) **24/40Ttaper Joint Clamp**
o) **Stainless Steel Ball-Joint Clamp**

9.2 **Allihn Condenser;** medium, Corning # 3480-MCO

9.3 **Constant Temperature Circulator;** VWR Scientific and Lauda (one unit each)

9.4 **Neslab Chiller;** CFT-33

9.5 **Graduated Cylinder;** 100 mL; Kimble # 20011-100 or equivalent

9.6 **Beakers;** assorted Pyrex 600 mL and 1000 mL, used for solvent containment and pipette storage.

9.7 **Glass Wool;** Silanized, Supelco #2-0410, solvent washed as per NE159.doc

9.8 **Glass Beads**

9.9 **Disposable Pasteur pipettes (9");** Krackeler #67-450-900-CS or equivalent

9.10 **pH Indicator Strips;** EM Science# 9590 or equivalent

9.11 **Microliter Gastight Syringes;** 500 and 1000 µL; Hamilton

9.12 **TurboVap Evaporator concentrator tubes (TurboTubes);** Zymark 250 mL, 0.5 mL endpoint.

9.13 **TurboVap Evaporator;** Zymark #ZW640-3

9.14 **Class A volumetric flasks with stoppers;** 5 and 10 mL; Pyrex #5640

9.15 **4 dram vials and 40 mL vials** with polyseal caps

9.16 **Vial rack;** plastic rack used to hold vials during all phases of the extract processing.

9.17 **Centrifuge;** International Equipment Co., Model CL (or equivalent)

9.18 **Wrist Shaker;** Burrel wrist action shaker, Model 75 and 88 (or equivalent)

9.19 **Auto sampler vials;** Scientific Resource Inc., # 99468-A or equivalent

10.0 Reagents and Standards

10.1 **1N Sodium Hydroxide;** J.T. Baker #5671-03, UN#1824 or equivalent

10.2 **Dichloromethane;** High purity solvent; Burdick/Jackson, #UN1593

10.3 **Hexane;** High purity solvent; Burdick/Jackson, #UN1208 or equivalent

10.4 **Acetone;** High purity solvent; Burdick/Jackson, #UN 1090 or equivalent

10.5 **Toluene;** High Purity Solvent; Burdick/Jackson, #UN1294 or equivalent

10.6 **1:1 Sulfuric Acid;** Mallinckrodt #2468, #UN1830, for pH adjustment (1:1 H_2SO_4 preparation: To a beaker containing 500mL cold DI water, slowly and under constant stirring, add 500mL concentrated H_2SO_4. Allow the mixture to cool after preparation.)

10.7 **Concentrated Sulfuric Acid;** Solvent washed as per NE174.doc, Mallinckrodt #2468, #UN1830 or equivalent

10.8 **Florisil;** J.T. Baker #M368-08. Solvent washed per NE094-05.doc
SEE SUPERVISOR FOR THE APPROPRIATE FLORISIL DEACTIVATION CONCENTRATION TO BE USED.

10.9 **Mercury;** Triple distilled, solvent washed as per NE175.doc, Mercury Waste Solutions Inc. (or equivalent)

11.0 Sample collection, Preservation, Shipment and Storage

The samples should be collected in unpreserved 1 liter glass bottles with Teflon lined cap. Samples should be delivered to the laboratory on ice and should be chilled to 4+-2 Degrees C. Samples are stored refrigerated at the laboratory at 4 ± 2 degrees C. The sample extraction hold time for this method is seven days from the date the samples were collected.

12.0 Quality Control

12.1 The extraction chemist should have completed an acceptable demonstration of precision and accuracy before performing the method without supervision. The addition of spiking material to a sample or blank must be witnessed by another extraction chemist and signed in the extraction logbook. All surrogates and matrix spikes must meet acceptable QC limits.

12.2 A method blank sample and lab control spike must be prepared per each extraction batch or 1 per 20 site samples whichever is more frequent. A matrix spike/matrix spike duplicate (or lab duplicate) should be prepared for every 20 site samples or as per client specified quality assurance project plan (QAPP). Spike defult for LCS, MS, MSD is 1.0mL of A1242 @ 0.5ppm. Client and/or project specifications may dictate alternate amount or Aroclor.

12.3 PCB Surrogates TCMX and DCBP are added to each sample prior to extraction to measure extraction/cleanup efficiency. Defult surrogate is: 1.0mL of 0.05ppm TCMX / 0.5ppm DCBP. Client and/or project specifications may dictate alternate amount.

13.0 Calibration and Standardization

Please see consult determinative methods (EPA Method 8082 (NE148.doc) and EPA Method 680 (NE040.doc) for Calibration and Standardization.

14.0 Procedure

14.1 SAMPLE PREPARATION

Note: Unless otherwise noted, when rinsing is referred to in this SOP, it will be with a Teflon squirt bottle. A solvent followed by a (b) will be used to designate solvent from a beaker.

14.1.1 Throughout the entire process it should be noted that if the extraction chemist encounters any problems or difficulties with any samples or steps involved, all work should **STOP!!!** Any problems should be brought to the attention of the supervisor and documented in the sample extraction logbook.

14.1.2 Before any steps are taken; the extraction chemist should first review the sample job folder and check the sample labels versus the original chain of custody. Any discrepancies should be noted in the sample extraction logbook.

14.1.3 Bring samples to room temperature by letting the samples warm up in the laboratory for a minimum of 30 minutes.

14.1.4 Mark the level of the sample on the outside of the sample container with a felt tip pen. Determine the pH of the sample by removing a small amount of sample with a disposable pipette (approximately 0.1 mL) and wet a pH indicator strip. The pH should be between 5 and 9. Adjust the pH with 1:1 sulfuric acid if the pH is greater than 9 or 5N sodium hydroxide if the pH is less than 5. Record the original pH in the extraction logbook.

14.1.5 Using 1 liter precleaned amber sample bottles, measure out two 1 liter aliquots of DI water to be used as an extraction blank and laboratory control spike.

14.2 ASSEMBLY OF PYREX ACCELERATED ONE STEP EXTRACTOR / CONCENTRATOR

See NEA233.SOP

14.3 EXTRACTION

14.3.1 **Observe sample:** In case of a high amount of sediment or very fine particles place glass wool and some glass beads on top of membrane assembly. This will prevent the obstruction of the membrane. **DO NOT** pack the glass wool down, otherwise you will obstruct the flow through the membrane.

14.3.2 Pour 55mL Dichloromethane (b) into extractor body. Check extractor for leaks which will most likely occur between the extractor body and cup assembly.

14.3.3 Add sample to the extractor unit. Rinse sample bottle with 55mL Dichloromethane (b) and add to extractor unit.

14.3.4 Add surrogate and matrix spike compound solutions directly into sample in the extractor body.

14.3.5 Place condenser into top of extractor body.

14.3.6 Open stopcock when both the chiller and constant temperature circulator have reached proper operating temperature. Solvent will begin to flow from extractor body into concentrator tube and immediately begin to boil.

14.3.7 Periodically verify that the boiling rate of the solvent is approximately 900mL per hour (once the unit is running and stabilized) as follows:

a) Note solvent level in concentrator tube.

b) Close stopcock and record how long it takes to boil 50mL of solvent over into extractor body.

c) The solvent should boil off in about 3 to 3.5 minutes, if the boiling rate is correct.

d) If **boiling rate** is too **slow**: Increase the temperature of the hot water circulator bath.
If **boiling rate** is too **fast**: Decrease the temperature of the hot water circulator bath.

14.3.8 Allow extraction to proceed for 2 hours.

14.3.9 Fill up sample bottle with water to previous level. Pour water into a 1 liter graduated cylinder and record sample volume in the extraction logbook. Discard sample container according to guidelines in Chemical Hygiene Plan.

14.4 SAMPLE EXTRACT CONCENTRATION

14.4.1 Close stopcock and allow solvent in concentrator tube to boil down.

14.4.2 Disconnect the concentrator tube from the hot water flow, when desired concentration volume of less than 10mL is reached. Sample will continue to boil while apparatus is cooling down. Sample extract will reflux from Snyder sidearm, which will increase the extract volume.

14.4.3 Attach the cold-water tubing to the concentrator tube.

14.4.4 Start cold water flow and cool for 10 to 15 minutes. Make sure extract is cold before disconnecting concentrator tube or loss of analytes may occur.

14.5 DISASSEMBLY

See NEA233.SOP

14.6 TRANSFER OF SAMPLE

14.6.1 Using a Pasteur pipette transfer the sample extract into a correctly labeled and Hexane pre-rinsed TurboTube or 40mL vial.

14.6.2 Rinse inside of concentrator tube with 3 Pasteur pipette volumes of Hexane and add to the sample extract. Repeat this step twice more. Rinse down the inside walls of the Turbo Tube. Add enough Hexane to the extract to get a total volume of approximately 100 mL in the TurboTube OR in the case of the 40mL vial add enough Hexane for a total of approximately 20 mL.

14.7 SOLVENT REDUCTION: TURBOVAP EVAPORATOR SYSTEM

14.7.1 The TurboVap evaporator system is used in place of the Kuderna Danish (KD) concentrator apparatus. The TurboVap evaporator system is used to reduce the sample volume. The TurboVap uses a heated water bath and positive pressure nitrogen flow with vortex action. The unit maintains a slight equilibrium imbalance between the liquid and the gaseous phase of the solvent extract, which allows fractional reduction of the solvent without loss of higher boiling point analytes.

14.7.2 Turn the unit on and allow it to heat up to 40º C ± 2º C.

14.7.3 As a precaution the TurboVap system regulators should be checked to insure that there is no residual gas pressure within the system and that the gas pressure regulator is off before placing samples in the apparatus. Residual gas pressure may cause splashing and therefore cause cross contamination of samples. To bleed the system of residual gas pressure, place an empty TurboTube into water bath and close lid. Make sure that the nitrogen gas pressure regulator is closed. Bleed any residual gas until the regulator gauge reads "0" psi. Remove the empty TurboTube.

14.7.4 If the sample has been transferred into a turbo tube proceed to 8.7.5. If the sample was initially stored in a 40mL vial, pour sample into a hexane pre-rinsed and correctly labeled TurboTube. To complete the transfer, rinse the threads with one disposable pipette volume of hexane. Rinse the 40mL vial with three pipette volumes of hexane. Add this to the TurboTube. Repeat this twice more. Rinse down the inside walls of the TurboTube. Bring the volume up to approximately 100 mL with hexane.

14.7.5 Add 200 uL of toluene to the sample extract (6-8 drops from a disposable pipette) for 8082 analysis. No toluene is added for 680 analysis.

14.7.6 Wipe down the inside of TurboVap with a hexane wetted paper towel including top lid and pins. Place TurboTubes containing the sample extract into TurboVap and close lid. Slowly open the pressure regulator. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the volume reduction.

14.7.7 The process for solvent (dichloromethane/hexane) reduction takes approximately 30 minutes. **DO NOT** leave the unit unattended as extracts may be blown to dryness and loss of PCB as well as surrogate and matrix spike may occur. Immediately notify the extraction supervisor if an extract is blown to dryness and note the incident in the sample extraction logbook.

14.7.8 Concentrate the solvent to approximately ½ the set volume (~2.5mL for samples to be set at 5.0ml), (5.0mL for samples to be set at 10mL). Remove the samples from the TurboVap and place into tube rack.

14.7.9 Using a disposable pipette quantitatively transfer the sample extract into a hexane pre-rinsed volumetric. Use hexane (b) to rinse the TurboTube and set to volume. Invert the volumetric with a prerinsed stopper several times to mix completely.

14.7.10 Transfer sample from the volumetric to a correctly labeled 4-dram vial .

14.7.11 All dirty glassware must be rinsed with acetone and dried in the fume hood before being washed with warm water, rinsed with RO water, and muffled.

14.8 SAMPLE EXTRACT CLEAN-UP PROCEDURE

Most extracts of environmental samples that are to be analyzed for PCBs contain co-extracted xenobiotics and other interfering substances which must be removed before accurate chromatographic analysis can be performed. Not all clean-up procedures need to be performed on every sample and several are sample matrix specific. The experience of the analyst combined with the sampling site history should guide the selection of which clean-up procedures are necessary. The extraction chemist records the sequence and number of repeats of cleanup steps performed in the sample logbook. Sample extract cleanups are performed on set volume extracts. The set volume is 5 or 10mL for water/wastewater samples.

14.8.1 Sulfuric Acid Wash

14.8.1.1 The concentrated sulfuric acid treatment removes hydrocarbons and other organic compounds which are co-extracted with the PCB residues.

14.8.1.2 Add one pipette volume full of solvent washed concentrated sulfuric acid (NE174.doc) to each extract vial and shake by hand in a fume hood for 30 seconds. Then centrifuge for at least 1 minute on setting #4. Transfer the hexane (upper) layer to a correctly labeled, pre-rinsed, 4-dram vial.

14.8.1.3 Repeat 8.7.1.2 if the sample extract appears to be heavily loaded (opaque) with colored material. Two to three acid washes may be required.
***Note*:** It is entirely possible that all colored material will not be removed from the extract.

14.8.2 Florisil Clean-Up

14.8.2.1 The florisil removes co-extracted polar compounds, residual water and residual sulfuric acid.
***Note*:** See supervisor for the appropriate florisil deactivation concentration to be used.

14.8.2.2 Add one spatula of florisil to each extract vial and shake by hand for 30 seconds. Swirl to get any florisil off the walls of the vial, then allow to settle. Transfer the hexane (upper) layer to a correctly labeled, pre-rinsed, 4-dram vial.

14.8.3 Removal of Sulfur Using Mercury

NOTE: Mercury is a highly toxic metal; all operations involving mercury should be performed within a fume hood. Prior to using mercury, the extraction chemist should become aquatinted with proper handling and emergency spill/clean-up procedures associated with this metal and must have reviewed the Material Safety Data Sheet (MSDS).

14.8.3.1 Add 1-3 drops of solvent washed mercury (NE175.doc) to the sample extracts and cap. Handshake for 15-30 seconds. If the mercury changes color, or breaks up into tiny balls and will not reform the original ball, change the mercury. To change the mercury transfer extract into a new correctly labeled and hexane pre-rinsed 4-dram vial and add new mercury to it. Repeat previous step. Place on wrist shaker for 30 minutes. The sulfur is converted to mercuric sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental sulfur.

14.8.3.2 Remove the sample extracts from the wrist shaker.

14.9 Final Extract Preparation

14.9.1 Transfer the extract to a hexane pre-rinsed and correctly labeled final 4-dram vial. Label with the client, test, dilution factor, date and sample ID number.

14.9.2 Transfer approximately 1.0 mL into a labeled 1.5 mL GC autosampler vial (8082 analysis only).

14.9.3 Submit the vials along with the project folder containing the Extraction Log and GC Queue Lab Sheet to the analyst.

15.0 Calculations

Please consult determinative methods (EPA Method 8082 (NE148.doc) and EPA Method 680 (NE040.doc) for calculations.

16.0 Method Performance

Please consult determinative methods (EPA Method 8082 (NE148.doc) and EPA Method 680 (NE040.doc) for Method Performance details.

17.0 Pollution Prevention

Please see NEA168.SOP for pollution prevention measures

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Please consult determinative methods (EPA Method 8082 (NE148.doc) and EPA Method 680 (NE040.doc) for Data Assessment and Acceptance Criteria for Quality Control Measures.

19.0 Corrective Action for Out-Of-Control Data

Please consult determinative methods (EPA Method 8082 (NE148.doc) and EPA Method 680 (NE040.doc) for Corrective Action for Out-Of-Control Data

20.0 Contingencies for Handling Out-Of-Control or Unacceptable Data

Please consult determinative methods (EPA Method 8082 (NE148.doc) and EPA Method 680 (NE040.doc) for Contingencies for Handling Out-Of-Control or Unacceptable Data

21.0 Waste Management

Please see NEA054.SOP, NEA083.SOP and NEA089.SOP

22.0 References

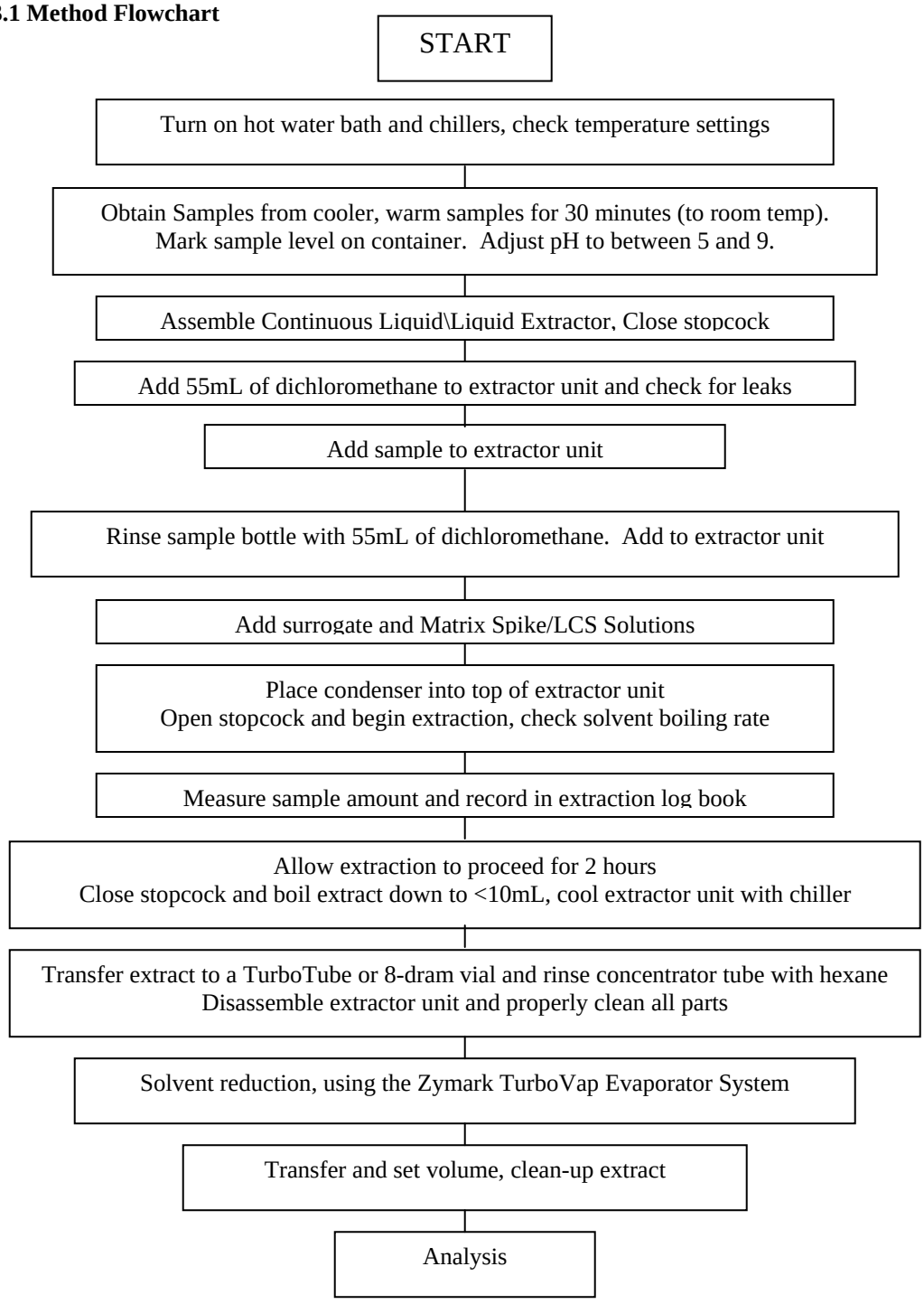
1. U.S. EPA SW-846 “Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods”, Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

2. “Guide to Environmental Analytical Methods”, Fourth Edition, Genium Publishing Corporation, 1998.

3. EPA Method 680 –EMSL Cincinnati, Ohio: Ann Alford-Stevens, Thomas A. Bellar, James W. Eichelberger, William L. Budde. November 1985.

23.0 Tables, Diagrams, Flowcharts and Validation Data

23.1 Method Flowchart



STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF PCB BY SW-846 METHOD 8082 WITH COMPREHENSIVE QUANTITATIVE CONGENER SPECIFIC ANALYSIS

NE133_02.SOP
Revision 02
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Reviewed and Approved By:

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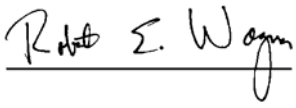
Laboratory Director:  Date: __12/08/2006__

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STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF PCB BY SW-846 METHOD 8082 WITH COMPREHENSIVE QUANTITATIVE CONGENER SPECIFIC ANALYSIS

1.0 Identification of Test Method

1.1 This method is used to determine polychlorinated biphenyls (PCBs) by high resolution gas chromatography with electron capture detection.

2.0 Applicable Matrix

2.1 This method is applicable in the determination of PCBs as described in EPA SW-846 Method 8082. It is applicable to the following matrices: water, soil, sediment, oil, fish, and tissue samples.

3.0 Detection Limit

3.1 Detection Limit: Reporting Limits (RLs) and Method Detection Limits (MDLs) vary for each matrix and are based on total PCB concentration.

3.2 MDL and RL are the following:

3.2.1 Aqueous Matrix: MDL 0.05ng/L-0.20ng/L per congener
RL 1.0ngL per congener

3.2.2 Solid Matrix: MDL 0.02ng/g-.15ng/g per congener
RL 1.25 ng/g per congener

3.3 Individual peak MDLs and RLs are determined every two years with matrix specific MDL studies. See Attachment A for MDL values.

4.0 Scope and application, including components to be analyzed

4.1 All 209 PCB congeners may be determined by this methodology. PCB congeners which exist at trace levels (<0.05 weight percentage) or undetected in Aroclor formulations (approximately 62 congeners) can be reported in this method. In environmental samples which are known to contain Aroclor based PCB contamination non-Aroclor congeners which co-elute with Aroclor congeners are assumed not to be present. Instrument calibration and calibration check standard preparations include all PCB congeners. Please see Table 1 for a complete listing of PCB congeners. There are 146 resolvable chromatographic peaks which provide complete separation for 99 of the congeners and coelution for the remaining 47 congeners. Of the 47 coelutions that occur, 20 are with non-Arolcor PCB congeners. See Attachment D for peak elution. This method is designed for the quantitation of total PCBs in samples resulting from both Aroclor type distributions and weathered, degraded, or non-Aroclor like distributions. Visual identification of Aroclor patterns (if present) may be possible in some cases by chromatographic comparison of samples with reference standards. A sum total of PCB present in the sample is provided by this method. This Method also allows estimation of homolog totals by level of chlorination.

4.2 In general, samples are extracted, or in the case of oils and waste solvent diluted, with a pesticide grade solvent. Applicable extraction methods for solids and animal tissues include: SW-846 Method 3540 (Soxhlet), SW-846 Method 3545 (Pressurized Fluid Extraction), or SW-846 Method 3550 (Ultrasonic Extraction). Extraction methods for aqueous samples include SW-846 Method 3510 (Separatory Funnel), and SW-846 Method 3520 (Continuous Liquid Liquid Extraction). The extracts are further processed by concentrating or diluting, depending on the PCB concentration, and carried through a series of clean-up

techniques. Applicable cleanup techniques include SW-846 Method 3620 (Florisil Cleanup), SW-846 Method 3660 (Sulfur Cleanup) and SW-846 Method 3665 (Sulfuric Acid/Permanganate Cleanup). The sample is then analyzed by direct injection onto a gas chromatographic system and detected by an electron capture detector.

4.3 This method provides detailed instructions for gas chromatographic conditions, calibration, and analysis of PCBs by capillary column gas chromatography. Each matrix requires different sample handling or special preparation procedure before analysis can be performed. Each sample matrix will be covered separately in the extraction standard operating procedures.

5.0 Summary of Test Method

5.1 Aroclor based PCB analysis methods (e.g. SW-846 8080 and 8082, EPA 608, and CLP methods) have been used for several years to measure total Aroclor concentrations in samples for regulatory compliance. In many situations these methods cannot accurately characterize the PCBs present in samples where environmental weathering and/or degradation by remediation (i.e. treatment) processes have occurred. In addition, samples containing mixtures of Aroclors may be difficult and or impossible to quantify using Aroclor based methodologies. In an attempt to address this problem techniques which determine the concentrations of individual PCB congeners in samples have been devised by the research community as well as the Environmental Protection Agency (EPA). SW-846 Method 8082 was written to provide analytical laboratories and EPA a defined analytical method which addresses some of the aforementioned problems. Method 8082 provides a framework for the measurement of both total Aroclor concentrations and individual PCB congener concentrations. Method 8082 allows flexibility in the specific instrumentation used as well as the total number of congeners analyzed and reported.

5.2 The purpose of this SOP is to provide a detailed written document for measurement of Polychlorinated Biphenyls (PCBs) according to SW-846 Method 8082 specifications with the addition of the Comprehensive Quantitative Congener Specific PCB Analysis (CQCS-PCB) developed by Northeast Analytical. The CQCS-PCB methodology provides the data user with a 146 peak assay of all 209 PCB congeners along with data reduction techniques which provide: Monochlorobiphenyl through Decachlorobiphenyl homolog distribution reported as weight percent and mole percent: Ortho, Meta, and Para chlorine/biphenyl distribution ratios; weight percent and mole percent values for each reported congener; individual concentration for each congener; total PCB concentration for the sample, congener identification by CAS#, IUPAC# and chlorine substitution pattern.

6.0 Definitions

6.1 Accuracy – The nearness of a result or the mean of a set to the true value. Accuracy is assessed by analysis of references samples and percent recoveries.

6.2 Analytical Batch –The basic unit for analytical quality control is the analytical batch, which is defined as samples which are analyzed together with the sample method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar matrices (e.g. water, sediment, soil, etc.).

6.3 Aroclor – Polychlorinated Biphenyls (PCBs) were commercially produced for a variety of uses including, transformers, capacitors, inks, paints, dust control agents, and pesticides. Monsanto Corporation was a major producer and sold PCBs under the trade name Aroclor.

6.4 Blank – A blank is an artificial sample designed to monitor the introduction of artifacts into the process. For aqueous samples, reagent water is used as a blank matrix, however, a universal blank matrix does not exist for solid samples, but sometimes sodium sulfate is used as a blank matrix. The blank is taken through the appropriate steps of the process. A reagent blank is an aliquot of analyte-free water or solvent analyzed with the analytical batch. Field blanks are aliquots of analyte-free water or solvents brought to the field is sealed containers and transported back to the laboratory with the sample containers. Trip blanks and equipment blanks are two specific types of field blanks. Trip blanks are not opened in the field. They are a check on sample contamination originating from sample transport, shipping and from site conditions. Equipment blanks are opened in the field and the contents are poured appropriately over or through the sample collection device, collected in a sample container, returned to the laboratory as a sample. Equipment blanks are a check on sampling device cleanliness.

6.5 Continuing Calibration Check Standard(CC) –The continuing calibration check standard contains all the target analytes found in the calibration standards and is used to verify that the initial calibration is prepared correctly and that the instrument system is correctly calibrated. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards (not independent for CQCS-PCB).

6.6 Calibration Standard (ICAL)– A series of known standard solutions used by the analyst for instrument calibration. Calibration standards are prepared from primary standard and/or stock standard solutions.

6.7 CAS Number – An assigned number used to identify a chemical. CAS stands for Chemical Abstracts Service, an organization that indexes information published in Chemical Abstracts by the American Chemical Society and that provides index guides by which information about particular substances may be located in the abstracts. Sequentially assigned CAS numbers identify specific chemicals, except when followed by an asterisk (*) which signifies a compound (often naturally occurring) of variable composition. The numbers have no chemical significance. The CAS number is a concise, unique means of material identification. (Chemical Abstracts Service, Division of American Chemical Society, Box 3012, Columbus, OH 43210: [614] 447-3600).

6.8 Congener – Any of the 209 Chlorinated Biphenyl compounds of varying degree of chlorination.

6.9 Duplicate– A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

6.10 Environmental Sample – An environmental sample or field sample is a representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination as requested or required. Environmental samples are normally classified as follows:

6.10.1 Drinking water -delivered (treated or untreated) water designated as potable water.

6.10.2 Water/Wastewater -raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluent.

6.10.3 Sludge -municipal sludges and industrial sludges.

6.10.4 Waste –aqueous and non-aqueous liquid wastes, chemical solids, contaminated soils, and industrial liquid and solid wastes.

6.11 Homolog – Any of the ten sets of PCB congeners of the same degree of chlorination.

6.12 Initial Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the analytical detector or method.

6.13 Instrument Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

6.14 Laboratory Control Sample (LCS) – Also known as the Quality Control (QC) Check Standard or Quality Control (QC) Check Sample. The LCS consists of an aliquot or reagent water or other blank matrix to which known quantities of the method analytes are added. The LCS is extracted and analyzed exactly like a field sample, and its purpose is to determine whether the analysis is in control and whether the laboratory is capable of making accurate and precise measurements.

6.15 Laboratory Method Blank – An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

6.16 Matrix – The predominant material of which the sample to be analyzed is composed. Matrix is not synonymous with phase (liquid or solid).

6.17 Matrix Spike – Aliquot of sample (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

6.18 Matrix Spike Duplicate – A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

6.19 Method Detection Limit (MDL) – The minimum constituent concentration that can be measured and reported with 99% confidence that the signal produced is different from the blank in a given matrix. The MDL is determined from a minimum of seven replicate samples, taken through the entire preparation and analysis procedure. The standard deviation, *s*, of those replicates is multiplied by a student's t factor in order to calculate the MDL.

6.20 MSDS – Material Safety Data Sheet. OSHA has established guidelines for the descriptive data that should be concisely provided on a data sheet to serve as the basis for written hazard communication programs.

6.21 PCB – Polychlorinated Biphenyl (PCBs) are a class of 209 individual chemical compounds (congeners), in which one to ten chlorine atoms are attached to biphenyl. Use of PCBs has made them a ubiquitous environmental pollutant.

6.22 Precision – The agreement between a set of replicate measurements without assumption of knowledge of the true value. Precision is assessed by means of duplicate/replicate sample analysis.

6.23 Quality Control – Set of measures within a sample analysis methodology to assure that the process is in control.

6.24 Standard Curve – A standard curve is a curve which plots concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by diluting the stock analyte solution in graduated amounts which cover the expected range of the samples being analyzed. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards must be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

6.25 Stock Solution – Standard solution which can be diluted to derive the other standards.

6.26 Surrogate – Organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, calibration and check standards, samples (including duplicates and QC reference sample) and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

6.27 Surrogate Standard – A pure compound added to a sample in the laboratory just before processing so that the overall efficiency of a method can be determined.

7.0 Interferences

7.1 One of the major sources of interference in the analysis of PCBs is co-extracted organochlorine pesticides. Several of the commonly found pesticides and associated pesticide degradation products (e.g. PP-DDT, PP-DDE, PP-DDD) overlap the PCB profile envelope and co-elute with several PCB peaks causing inaccurate measurement. The analyst must be careful to review the chromatographic pattern and peak retention times and flag peaks that are suspected of containing interference so that they are not included in the total PCB values generated. Rigorous cleanup of sample extract (i.e. sulfuric acid cleanup and florisil cleanup removes many non-target pesticides).

7.2 Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing. Samples and extracts should not be exposed to plastic materials. Phthalate esters exhibit response on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Laboratory method blanks must be thoroughly reviewed for presence of non-target peaks and comparison of samples with blank chromatographic patterns.

7.3 The sample matrix itself is also a potential source for method analyte interference. Sample preparation, extraction procedures, and extract clean-up protocols are covered in separate SOPs that deal exclusively with sample extraction.

8.0 Safety

8.1 Safety glasses and disposable gloves must be worn when handling samples and extracts.

8.2 All manipulations of sample extracts should be conducted inside a chemical fume hood. Manipulation of sample extracts outside of a fume hood should be minimized by the analyst.

8.3 Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets to familiarize themselves with the precautions for handling solvents and chemicals used to process samples. The analyst should refer to the laboratory chemical hygiene plan for further safety information.

8.4 Samples remaining after analysis should either be returned to the customer for disposal or disposed of through the laboratory's disposal plan. Refer to the sample custodian for assistance and also standard operating procedure NEO54, disposal of laboratory waste.

9.0 Equipment and Supplies

9.1 Gas Chromatograph: Complete system for high resolution, capillary column capability and all required accessories. Northeast Analytical, Inc. will use an Agilent Model 6890 gas chromatograph (or equivalent), equipped with capillary split/splitless injector (or equivalent), temperature programmable oven, Model 7683 automatic sampler (or equivalent), and micro-electron capture detector (or equivalent). A data system and integration of detector signal is interfaced to the gas chromatograph.

9.2 Chromatographic Data System: A data system for measuring peak height and peak area. An Empower computer network based workstation (Waters Corporation), will be employed to capture detector response and digitally store the chromatographic, electronic peak integration for precise calculations, database structuring of the analytical information, and archival capabilities.

9.3 Gas Chromatographic Column: Chrompack CP-Sil5/C18, 50 meter, 0.25mm ID, 0.10 micron phase thickness, Cat. No 7477.

9.4 Class A volumetric flasks: 5.0 – 100mL.

9.5 8 dram vials and 4 dram for sample extract storage.

9.6 Pasteur pipettes.

9.7 250ml and 100ml beakers, glass.

9.8 Disposable 1.0, 5.0, and 10.0 ml pipettes.

9.9 Hexane, Burdick and Jackson.

9.10 Acetone, Burdick and Jackson.

9.11 Toluene, Burdick and Jackson.

9.12 Methylene Chloride, Burdick and Jackson.

9.13 Ferrules: 0.4mm graphite/vespel, Aglient 1858 or equivalent.
9.14 Injector septa: Restek Ice Blue Septa (22392) or equivalent.

9.15 Injector liner: Restek 4mm Split Liner for Varian GC's (21046) or equivalent.

9.16 Viton Fluorocarbon O-ring: Restek (20377) or equivalent.

9.17 Hamilton Injector Syringe 5.0 µL: Hamilton 87994

9.18 Auto sampler vials: 12X32mm Clear R.A.M. Marking Spot Vial Black Closure w/TFE Septa (Cat. No. 402105-540).

10.0 Reagents and Standards

10.1 Preparation of PCB congener Stock Standard Solution and 1,2,3,4,5,6,7,8-Octachloronaphthalene stock solution.

10.1.1 Primary Stock Polychlorinated Biphenyl Congener Solutions: Accustandard Cat. No. C-CSQ-SET calibration mixtures contains all 209 PCB Congeners. The primary stock standard is prepared by combining 1-9 of the primary stock congener mixes. Each mix has a concentration of 10.0ug/ml. Mix 0.500ml of each congener mix (1-9) into 10ml volumetric flask and bring it up to 10ml with hexane to form a single solution at 500ng/ml. To produce the secondary stock solution one would mix 1.0ml of the primary stock standard into a 10ml volumetric flask and bring it up to 10ml with hexane to form a single solution at 50ng/ml.

10.1.2 Primary Stock 1,2,3,4,5,6,7,8-Octachloronaphtalene (OCN) solutions (internal standard): Accustandard Cat. No. N-003N. The primary stock standard for OCN is prepared by weighing out 10.1mg of the stock standard which contains 25mg of OCN. Quantitatively transfer 10.1mg into 50.0ml of Toluene (may take a view minutes for OCN to go into solution). This will produce a solution concentration of 202ug/ml. To produce the secondary stock solution one would mix 1.0ml of the primary stock standard into a 10ml volumetric flask and bring it up to 10ml with toluene to form a single solution at 20.2ug/ml.

10.1.3 The internal standard will be added to calibration standards, sample, extracts, blanks, and QC samples prior to gas chromatographic analysis. Thus, the internal standard is used as a quantification spiking standard and will eliminate sample injection volume variations, but will not correct for analytical loses during sample preparation.

10.1.4 The stock standards are transferred into screw-cap vials and stored in a freezer, protected from light. Stock standards should be checked frequently for signs of evaporation, especially just prior to preparing calibration standards. Stock PCB standards must be replaced after on year, or sooner if a problem with instrument calibration is detected.

10.2 Surrogate Stock Standards

10.2.1 Surrogate stock standard (2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl) at 100ug/ml: The surrogate stock standard is prepared from a solid standard 2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl, Accustandard Cat. No. C-207N. Weigh 5.0mg of the solid surrogate standard into a solvent rinsed 10ml vial. Quantitatively transfer the surrogate standard using ten successive 2-ml washings of hexane to a 50ml volumetric flask. Be sure to rinse the 10ml vial wall carefully so that the entire surrogate standard is completely transferred to the 50ml volumetric flask. Make to volume with hexane and mix the surrogate standard solution by shaking the flask several times. This will give a concentration of surrogate standard of 100ug/mL. Carefully transfer the surrogate standard solution to 125ml boston bottle, tightly cap, and store in a freezer or refrigerator. The surrogate standard must be replaced after one year.

10.3 Calibration Standards

10.3.1 Calibration standards are prepared at five concentration levels using the high level 50ng/ml stock standard and the 500ng/ml commercial stock standard. The following five standards make up the initial calibration curve standard set: 0.100ng/ml, 0.500ng/ml, 5.0ng/ml, 25ng/ml, and 50ng/ml. Octachloronapthalene (OCN internal standard) is added to each calibration standard at 20.2ug/ml to be used as an internal standard.

10.3.2 <u>50ng/ml Calibration Standard with OCN at 18.18ng/ml</u> – The 50ng/ml calibration standard is prepared from the 500ng/ml commercial stock standard and the 20.2ug/ml OCN internal standard. Into a 10ml volumetric flask, using a 10-microliter syringe, transfer 9.0ul of the 20.2ug/ml OCN internal standard. Into the same 10ml volumetric flask, using a 1000-microliter syringe, transfer 1000ul of the 500ng/ml primary stock standard. Make to volume with hexane and transfer the standard solution to a 4-dram vial, tightly cap, and store in the refrigerator. This will give a concentration of 50ng/ml for the congener standard and a concentration of 18.18ng/ml for the internal standard (OCN),

10.3.3 25ng/ml Calibration Standard with OCN at 18.18ng/ml – The 25ng/ml calibration standard is prepared from the 50ng/ml secondary stock standard and the 20.2ug/ml OCN internal standard. Into a 10ml volumetric flask, using a 10-microliter syringe, transfer 9.0ul of the 20.2ug/ml OCN internal standard. Into the same 10ml volumetric flask, using a 5.0ml Class A pipette, transfer 5.0mls of the 50ng/ml secondary stock standard. Make to volume with hexane and transfer the standard solution to a 4-dram vial, tightly cap, and store in the refrigerator. This will give a concentration of 25ng/ml for the congener standard and a concentration of 18.18ng/ml for the internal standard (OCN).

10.3.4 5.0ng/ml Calibration Standard with OCN at 18.18ng/ml – The 5.0ng/ml calibration standard is prepared from the 50ng/ml secondary stock standard and the 20.2ug/ml OCN internal standard. Into a 25ml volumetric flask, using a 50-mircroliter syringe, transfer 22.5ul of the 20.2ug/ml OCN internal standard. Into the same 25ml volumetric flask, using a 2.5ml syringe, transfer 2.5mls of the 50ng/ml secondary stock standard. Make to volume with hexane and transfer standard solution into an 8-dram vial, tightly cap, and store in the refrigerator. This will give a concentration of 5.0ng/ml for the congener standard and a concentration of 18.18ng/ml for the internal standard (OCN).

10.3.5 0.50ng/ml Calibration Standard with OCN at 18.18ng/ml – The 0.50ng/ml calibration is prepared from the 50ng/ml secondary stock standard and the 20.2ug/ml OCN internal standard. Into a 10ml volumetric flask, using a 10-microliter syringe, transfer 9.0ul of the 20.2ug/ml OCN internal standard. Into the same 10ml volumetric flask, using a 100-microliter syringe, transfer 100uls of the 50ng/ml secondary stock standard. Make to volume with hexane and transfer standard solution in a 4-dram vial, tightly cap, and store in the refrigerator. This will give a concentration of 0.50ng/ml for the congener standard and a concentration of 18.18ng/ml for the internal standard (OCN).

10.3.6 0.10ng/ml Calibration Standard with OCN at 18.18ng/ml- The 0.10ng/ml calibration is prepared from the 50ng/ml secondary stock standard and the 20.2ug/ml OCN internal standard. Into a 10ml volumetric flask, using a 10-microliter syringe, transfer 9.0ul of the 20.2ug/ml OCN internal standard. Into the same 10ml volumetric flask, using a 50-microliter syringe, transfer 20ul of the 50ng/ml secondary stock standard. Make to volume with hexane and transfer standard solution in a 4-dram vial, tightly cap, and store in the refrigerator. This will give a concentration of 0.10ng/ml for the congener standard and a concentration of 18.18ng/ml for the internal standard (OCN).

10.3.7 Continuing Calibration Check Standard @5.0ng/ml with OCN at 18.18ng/ml(CC) – The 5.0ng/ml calibration check standard is used as the continuing calibration check standard. No independent reference material is available at this time.

10.3.8 Calibration standards must be replaced after one year, or sooner, if a problem is detected.

11.0 Sample Collection, Preservation, Shipment and Storage

11.1 Sample Collection and Preservation:

11.1.1 Routine soil, sediment, sludge, solid and concentrated liquid samples should be collected in 8 oz clear glass wide-mouth jars, fitted with a Teflon-lined cap. Aqueous samples should be collected in 1 liter amber glass bottles with a Teflon-lined cap. Project specific, the jars maybe required to be pre-cleaned to EPA specification protocol A – recommended for extractable organic, semivolatile and pesticide analysis. Protect samples from light.

11.1.2 All samples must be placed on ice or refrigerated at 4°C (± 2°C) from the time they are collected until delivery to the lab.

11.2 Sample Shipment:

11.2.1 Sample Shipment is accomplished through a carrier such as Federal Express or United Postal Service for overnight 1-day delivery to the lab. Shipment is normally handled by the field personnel collecting the samples and coordinated with sample receiving department at the lab. Samples can also be picked up by the lab courier service if samples are collected within driving distance to the lab.

11.3 Sample Storage:

11.3.1 The samples must be protected from light and refrigerated at 4°C (± 2°C) from time of receipt until they are removed from storage for extraction. Remaining sample material will be stored protected from light and refrigerated at 4°C (± 2°C). Sample will be disposed of or stored / archived according to project specifications.

11.3.2 Routine soil, sediment, sludge, solid, liquid and concentrated liquid samples are stored in a refrigerator dedicated for this type of sample.

11.4 Sample Extract Storage:

11.4.1 Sample extracts must be protected from light and stored refrigerated at 4°C (± 2°C) during the analysis. After analysis is complete, sample extracts will be discarded after 60 days or can be archived in a freezer at less than -20°C for longer periods of time depending on the program requirements.

11.4.2 Field samples, sample extracts, and calibration standards must be stored separately.

11.5 Required Hold Time

11.5.1 Extraction of solid samples by appropriate technique must be completed within fourteen days from sample collection.

11.5.2 Extraction of aqueous samples by appropriate technique must be completed within seven days from sample collection.

11.5.3 Sample extracts must be analyzed within forty days of sample extraction.

12.0 Quality Control

12.1 This section outlines the necessary quality control samples that need to be generated at the time of sample extraction. The results of the quality control measurement samples document the quality of the data generated. The following table lists the Quality Control samples required for capillary gas chromatography analysis of PCBs.

Quality Control Requirements

QC Sample	Frequency
Method Blank	With each sample batch (up to 20 samples)
Lab Control Spike	With each sample batch (up to 20 samples)
Cont Cal Check Std	Analyzed prior to each sample batch and at a Frequency or one per ten injections. Each analytical sequence must close with a Continuing Calibration Check Standard (CC).
Duplicate Analysis	Field generated sample – analyzed at discretion of client.
Matrix Spike	One matrix spike per 20 field samples or designated sample batch may be performed as specified in the client site plan.
Matrix Spike Duplicate	One matrix spike duplicate per 20 field samples or designated sample batch may be performed as specified in the client site plan.

12.2 Method Blank

12.2.1 With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For water samples and organic-free reagent water blank is processed. For soil, sediment, solid, fish, and other tissue sample a laboratory clean sodium sulfate is processed. For oil samples, a PCB-free blank control oil is processed. The method blank must exhibit PCB levels less than the matrix defined reporting limit (RL). If the method blank exhibits PCB contamination above the reporting limit, the samples associated with the contaminated blank should be re-extracted and analysis repeated. If there is no original sample available for re-extraction, then the results should be flagged with a "B" indicating blank contamination. The value measured in the blank is reported for those samples associated with the particular blank out of criteria.

12.3 Laboratory Control Spike

12.3.1 A Laboratory Control Spike (LCS), also referred to as a QC reference check standard, is extracted with each batch of samples at a rate of one per 20 samples. For water sample, spike one liter of laboratory organic free water, extract and analyze. For solid and tissue samples spike 10 grams of sodium sulfate, extract and analyze. For oil samples spike 1 gram of PCB free oil, extract and analyze. An Aroclor is chosen for the LCS analyte, typically based on program requirements or expected sample contamination. Calculate the percent recovery for the PCB spike. If the percent recovery for the LCS is out of criteria, (60%-140%) the analysis is out of the control for that analyte and the problem should be immediately corrected.

12.4 Duplicate Analysis

12.4.1 Duplicate analysis of the same sample is performed to assess method precision. A duplicate can also be performed as a blind duplicate, so that identification with original sample is withheld. The analysis of a duplicate sample precludes that PCBs are to be found at appreciable levels in samples. If this is not known the analysis of matrix spike/matrix spike duplicates provide more consistent quality control information. The relative percent difference of the two measurements on the sample is calculated on total PCB concentration by the following equation:

$$RPD = (DUP1-DUP2)/AVG \times 100$$

Where: RPD = Relative Percent Difference
DUP1 = The greater of the measured values
DUP2 = The lesser of the measured values
AVG = Average of the two analysis

The relative percent difference must be less than or equal to 30%.

12.5 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

12.5.1 Spiked sample matrix data are analyzed to assess analytical accuracy and recovery of analytes of interest. Thus the sample is spiked and carried through sample analytical procedures including extraction, clean up, and GC analysis. Depending on the specific project plans and at the discretion of the client a matrix spike or matrix spike and matrix spike duplicate can be analyzed.

12.5.2 There must be sufficient sample for analysis of matrix spike/matrix spike duplicate samples and the sample must be homogeneous in PCB distribution for valid data to be produced. Spike MS and/or MSD samples with the Aroclor matrix spike standard at a concentration approximately two to five times the sample concentration. Extract and analyze the two spiked samples following procedures used for actual sample analysis. Calculate the percent recovery of the matrix spike/matrix spike duplicate by the following equation:

$$P = A-B/T \times 100$$

Where: P = Percent recovery, %
A = concentration of analyte in the spike sample aliquot
T = Know true values of the spike concentration
B = Background concentration of PCB in the unspiked sample aliquot

12.5.3 Matrix spike recovery information is used to assess the long-term precision and accuracy of the method for each encountered matrix. Matrix spike/matrix spike duplicate results are not used alone to qualify an extraction batch. Generally, percent recovery for MS/MSD samples should be greater than or equal to 60% and less than or equal to 140% based on the total PCB concentration. If the percent recovery is outside the limits, all calculations should be checked and the data should be narrated to describe possible matrix interference.

12.6 Surrogates:

12.6.1 Surrogate-spiking compounds monitor the extraction efficiency and sample processing procedures for each sample. Surrogate compounds are chosen which do not chromatographically interfere with the PCB target congeners and which behave similarly to the target PCB congeners during extraction and sample processing.

12.6.2 A surrogate compound is added to each sample, matrix spike, matrix spike duplicate, duplicate, method blank, and laboratory control spike at the time of extraction. The surrogate compound chosen for this method is 2,2',3,3',4,4',5,6,6' – Nonachlorobiphenyl, Accustandard Cat. No. C-206S-TP (this congener is not present or found at trace amounts in Aroclor formulations). The following are typical surrogate amounts added to normally encountered matrices. These amounts can be adjusted by the analyst, if PCB background levels are high and surrogates are being diluted out of analysis range.

12.6.2.1 Water: 1.0ml of 1.0ppm 2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl

12.6.2.2 Soil/Sediment/Solid/Fish: 0.400ml of 1.25ppm 2,2',3,3',4,4',5,6,6'-Nonachlorobipenyl.

12.6.3 Surrogate recovery measurements are used for advisory purposes, if surrogate recovery is not within laboratory establish limits, the following steps are required. The laboratory established limits for surrogate recovery are 60%-140%.

12.6.5.1 Review calculations that were used to generated surrogate percent recovery values to make certain there are no errors.

12.6.5.2 Check by GC analysis surrogate solutions used during sample extraction steps to ensure that no problems exist with spiking solutions.

12.6.5.3 Review data for chromatographic interferences.

12.6.5.4 Re-extraction and/or re-analysis of samples may be indicated if problems persist with surrogate recoveries. If the surrogate percent recovery is out of limits on the re-extracted samples, low or high surrogate recovery is due to matrix affects and the data can be reported as estimated.

12.7 Continuing Calibration Check Standard (CC)

12.7.1 To begin an analytical sequence the calibration check standard containing each congener at 5ppb is injected and analyzed. If more than 12 hours has elapsed since the last valid continuing calibration check standard another standard must be injected before beginning an analytical sequence. If less than 12 hours has elapsed since the last valid continuing calibration check standard, analysis of samples may begin, a continuing calibration check standard must however be analyzed after each 10 sample injections.

12.7.2 The calibration factor for the continuing calibration check standard must be less than 15% difference for it to be valid and analysis to proceed. If this criterion is exceeded, the analyst should inspect the system to determine the cause and perform maintenance as necessary. The system can then be recalibrated and sample analysis can proceed. Note that all samples injected after the standard exceeding the 15% difference criterion must be re-analyzed when the system is in-control.

12.8 Retention Time

12.8.1 The retention time (RT) windows are established from the Continuing Calibration Check Standard (CCS) peak retention times. The CCS is analyzed three times over a 72-hour period and the standard deviation is calculated from the three retention time measurements. The standard deviation is multiplied by three and this establishes the retention time window for each quantified peak (±3SD). Use the retention time for a peak in the continuing calibration check standard to determine the mid point of the retention time window for the analysis sequence. Each time a CCS is analyzed the retention time windows should be updated. This function is performed in the chromatography software graphically as vertical dropdown retention time markers with retention time window brackets. Besides using the retention time window to assign peaks for quantification, the analyst should also rely on their experience in pattern recognition of multi-response sample analysis.

12.9 Analytical Sequence Queue:

12.9.1 A typical analytical sequence is as follows:

Injections	Material Injected
1-2	Hexane Instrument Blanks
3-6	Initial Calibration Standards
7	Continuing Calibration Check Standard
8	Method Blank
9	Lab Control Spike
10-18	Samples (including Duplicates, MS/MSD)
19	Continuing Calibration Check Standard
20-28	Samples (including Duplicates, MS/MSD)
29	Continuing Calibration Check Standard

13.0 Calibration and Standardization

13.1 Gas chromatographic operation parameters:

13.2 Establish the gas chromatographic operation parameters as follows:

13.2.1 GC Column: Chrompack CP-Si15/C18, 50 meter, 0.25mm internal diameter, 0.10 micron phase thickness, Cat No. 7477.

13.2.2 Oven Temperature Program: 50°C for 1.0min hold time, 50°C to 150°C at 15.0°C/min, hold 0.01 minutes, 150°C-250°C at 0.80°C/minute, hold at 250°C for 2.40 minutes.

13.2.3 GC Column Velocity: Approximately 33 cm/sec Helium. Column flow adjusted to elute OCN Internal Standard between 126 and 130 minutes.

13.2.4 Detector: Electron Capture Detector (micro-ECD), attenuation 0, range 3, (adjustable based on signal sensitivity). Detector temperature at 300°C.

13.2.5 Injector Temperature Program: 275°C, pulsed splitless injection, initial pressure at 25.0PSI, pulsed pressure at 50PSI, purge time 0.50 minutes.

13.2.6 Detector Make-up Gas: Approximately 60mL/min Nitrogen. Adjusted for signal sensitivity.

13.2.7 Autosampler: 1.0ul sample volume (adjustable based on signal sensitivity). Sample pumps 3, viscosity delay 0, sample wash 1, solvent A 2 washers, solvent B 2 washes, slow plunger OFF, sampling offset OFF, solvent A pre-wash 1, solvent B pre-wash 1.

13.3 Initial GC Calibration

13.3.1 GC calibration is performed by the internal standard calibration procedure. Prior to running samples the system must be calibrated and system performance must be verified.

13.3.2 Establish the gas chromatographic operating parameters outlined in the Procedure section and prepare the calibration standards at the four concentrations outlined in the Reagent and Standard section. Inject each calibration standard using the GC Autosampler and the parameters outlined in the Procedure section. Note: The same parameters are used for actual samples.

13.3.3 Attachment B provides data on a typical calibration analysis including percent relative standard deviation of the calibration factors.

13.3.4 For the initial calibration curve to be considered valid, the percent relative standard deviation of response factors must be less than 20% over the working range. The calibration curve is used for quantitation in every case and is not replaced with the average calibration factor.

13.3.5 Our laboratory uses a computer based chromatography software module (Water Corporation, Empower software) interfaced to the gas chromatograph. The workstation processes the detector signal, performs an analog to digital conversion, and stores the digitized chromatograms on the computer hard disk. Integration of peak areas and production of chromatograms is performed in the Empower software. All data analysis will be carried out on specialized software developed at Northeast Analytical including calculating calibration curves/response factors, report generation, and archival of data. The data is transferred to the in-house software and response factors are calculated for each separated and identified peak.

13.3.6 The percent recovery for the internal standard octachloronaphthalene (OCN) in the CC must be within 50-150% of the OCN area in the associated calibration standard.

13.3.7 If a re-calibration is performed, the CC must be analyzed again and values calculated using the new relative response factors. If the CC fails to meet the percent difference criteria after re-calibration, sample analysis must not proceed until the problem is found and corrected (*i.e.*, GC gas leak, autosampler lines plugged, broken injector liner).

13.4 Retention Time Windows

13.4.1 To establish retention time windows the GC system should be checked by the analyst to make sure it is functioning properly before establishing retention time windows. Make three injections of one selected concentration level calibration standard containing each PCB congener through out a minimum 72-hour time period.

13.4.2 For each peak calculate the standard deviation resulting from the variation in the three retention times for that peak. If the standard deviation of the selected peak is zero, the standard deviation of the peak eluting after it is used. If it is the last eluting peak that has zero for the standard deviation, then substitute the standard deviation of the peak eluting before the last peak. Attachment C includes an example of calculated retention time windows generated by the procedures outlined above.

14.0 Procedure

14.1 Sample Extraction and Preparation

14.1.1 The following SOPs detail sample extraction procedures that are utilized in preparing samples for analysis by this analytical method:

SOP NAME	TITLE
NE005	Soxhlet Extraction : Solids
NE006	Water Extraction
NE017	Fish & Biota Extraction
NE049	Animal Tissue Extraction: Small Mass Procedure
NE087	Extraction for Oil
NE088	Wipe Extraction for PCB
NE142	CLLE PCB Water Extraction
NE132	Fish/Biota Grinding Procedures
NE140	PCB Screening by GC
NE143	ASE Extraction for PCB: Solids
NE144	ASE Extraction PCB: Wipes
NE158	% Lipid Determination: Fish & Biota

14.2 Gas Chromatographic Procedures

14.2.1 Prescreening of sample extracts: See standard operating procedure NE140 for details on the PCB screening procedures used prior to final analysis by this method. The GC will be standardized by using Aroclor 1221, Aroclor 1242, and Aroclor 1260. These three Aroclor formulations incorporate most environmental PCBs found in sample extracts and provide a good estimate of PCB amount for final dilution for capillary analysis. A three level calibration curve is utilized (0.50ug/ml, 2.5ug/ml, and 5.0ug/ml standards). The concentration of each Aroclor (grouped as Aroclor 1221, Aroclor 1242, and Aroclor 1260 only) in a sample will be calculated based on the extract volume (not the sample weight or volume) to supply solution concentration values that show if the extract needs to be diluted for final capillary GC analysis. If a dilution is necessary, sample extracts are diluted to a solution concentration between 0.250ug/mL-0.300ug/mL depending of the Aroclor pattern from the pre-screening results. Preferable solution set volumes are 5ml and 10ml as the internal standard (OCN) is added at the time to give a concentration of 0.1818ug/ml in the final dilution.

14.2.2 Approximately 1.0ml of the final dilution extract is then transferred into a labeled autosampler vial.

14.2.3 The sequence of the analytical queue is set up in the NEA LIMS as a unique batch file. This file contains the exact order in which standards, instrument blanks, and samples will be analyzed. Once the sample set is uploaded into the Empower acquisition/run screen and saved, the sample set is printed and the samples are loaded into the GC autosampler tray in the order specified by the sample set queue.

14.2.4 The following labeling will be used on the autosampler vial and for the sample set file createdfor the analytical queue.

14.2.4.1 The initial calibration standard will be labeled as ICAL110306A, ICAL110306B, etc. Substitute the actual date of analysis in the file name.

14.2.4.2 The instrument blanks will be labeled 061103B01,B02,B03, etc. Substitute the actual date of analysis in the file name.

14.2.4.3 The Continuing calibration check standards will be labeled CCS1103A,CCS1103B, etc. Substitute the actual date of analysis in the file name.

14.2.4.4 Samples are labeled with the laboratory identification number on the autosampler vial. In the sample set file the laboratory identification number, along with the client identification, sample weight, set volume and dilution are entered.

14.2.5 At this point the chromatography software can be initiated to start data collection. The gas chromatograph is placed into run mode and sample analysis is performed until the analytical queue is complete.

14.2.6 Peak Identification

14.2.6.1 Target peaks are identified in unknown samples based upon Retention Time (RT). The retention time of an unknown peak must fall within the retention time windows established. Attachment C is an example of peak identification based on retention time.

14.2.6.2 Besides using retention time windows to assign peak IDs, the analyst should also rely on their own experience in pattern recognition of multi-response PCB chromatograms. Caution should be exercised when identifying peaks which elute near interferences present in samples and blanks. Comparison of sample chromatograms with method blank and field blank chromatograms is useful in determining chromatographic interferences.

14.2.6.3 This method should be applied with caution when used to determine individual congeners of interest in unknown sample for which no prior historical information exists. In this case confirmatory column analysis or confirmation by GC/MS analysis may be advised.

14.3 Data Reduction/Reporting

14.3.1 The data file (text format) obtained is imported into the in-house software and processed. Final peak assignments and quantitation calculations are performed within the software along with the current instrument calibration. Peaks which are known to represent co-eluting congeners are mathematically quantitated as individual congeners using established ratios. The final concentration results are provided in the reporting section of the software. The following information is provided:

14.3.1.1 Monochlorobiphenyl through Decachlorobiphenyl homolog distribution reported as weight percent and mole percentage.

14.3.1.2 Ortho, Meta and Para chlorine/biphenyl distribution ratios.

14.3.1.3 Weight percent and mole percent values fro each reported congener.

14.3.1.4 Individual concentration for each congener.

14.3.1.5 Total PCB concentration for the sample.

14.3.1.6 Congener identification by CAS#, IUPAC#, and chlorine substitution pattern.

14.3.1.7 Surrogate percent recovery

15.0 Calculations

15.1 Screening GC: External Standard Calibration

15.1.1 The GC screening analysis will be done by the external standard calibration technique. See standard operating procedure NE140 for details on PCB screening procedures used prior to final analysis by this method. The calibration curves for each section of the PCB elution profile will be calculated using the following formula:

$$\text{Calibration factor} = \frac{\text{Amount (ug) of Aroclor`}}{\text{Total area of Aroclor}}$$

15.1.2 The calibration curve will be a linear fit forced through zero.

15.2 Screening GC: Sample Calculations:

15.2.1 The concentration of each Aroclor (grouped as Aroclor 1221, Aroclor 1242 and Aroclor 1260 only) in a sample will be calculated based on the extract volume (not the same weight or volume) to supply solution concentration values that show if the extract needs to be diluted for final capillary GC analysis. The solution concentration of Arolcor 1221, Aroclor 1242, or Aroclor 1260 (or all 3) in a sample is calculated as follows.

Concentration (ug/mL) = (Ax) x (CF)
Where:
Ax = Area of Aroclor of interest in sample
CF =Calibration factor in standard

15.3 Capillary GC: Internal Standard Calibration

15.3.1 The capillary column GC analysis will be done by the internal calibration technique. Calibration and sample quantification will be performed by a commercial GC software package. The capillary GC will be standardized by using a calibration standard that contains all 209 PCB congeners.

15.3.2 Relative Response Factors for each separated and identified peak in the standard will be calculated using the following formula:

RRF = (Ax/Ais)x(Cis/Cx)
Where:
RRF = Relative response factor of congener(s).
Ax = Area of peak for the congener(s).
Ais = Area of peak for the internal standard.
Cx = Concentration of the congener(s).
Cis = Concentration of the internal standard.

15.4 Capillary GC: Sample calculations

15.4.1 The concentration of each identified PCB peak in a sample will be calculated based on the sample dry weight in the case of soils, sediment, and wet weight for fish and biota samples.

15.4.2 The sample PCB concentration of each standardized PCB peak is calculated as follows:

$$\text{Concentration (ng/g)} = \frac{[(Ax)(Cis)(V)(D)]}{[(Ais)(RRF)(Ws \text{ or } Ls)]}$$

Where:
Ax = Peak area for congener(s) being measured.
Cis = Amount of internal standard added to sample extract.
D = Dilution factor, if sample was diluted prior to analysis.
V = Extract volume.
Ais = Peak area of added internal standard
RRF = Relative response factor for congener(s) being measured.
Ws = Dry or wet weight of sample.
Lx = Volume of sample extracted

15.5 The calculated PCB concentration for each PCB peak will be compared to its respective sample-specific reporting limit (RL) and method detection limit (MDL). The results for peaks with concentrations at or above the MDL but below RL will be reported as detects and flagged as estimated J. The results for peaks with concentrations at or above the RL would be reported as unqualified numeric values.

16.0 Method Performance

16.1 Precision and Accuracy Determination

16.1.1 Precision and accuracy data is obtained for the method by analyzing four laboratory control spikes at a reasonable concentration below the high calibration standard and above the low calibration standard. The analyte will be added to a laboratory organic free water sample or sodium sulfate and taken through all extraction and analytical procedures. Aroclor 1242 is used as the analyte and recovery on a total basis is used to calculate percent recovery. To be valid, Aroclor 1242 will be recovered between the limits of 70 to 130 percent. Also, a

percent relative standard deviation will be calculated for the replicates and will be less than or equal to 20% to be valid.

16.2 Method Detection Limit

16.2.1 A method detection limit will be determined for this method whenever major modification to the extraction or analysis procedures are made or at a minimum frequency of every 2 years. A minimum of seven laboratory organic free water samples or sodium sulfate will be prepared and spiked with the complete PCB congener calibration standard (all 209 PCB congeners). at a low level and taken through all extraction and analytical procedures. Method detection limit data will be determined for each chromatographic peak (comprising one or more PCB congeners) based on the following equation:

$MDL = S * t_{(n-1, 1-alpha=0.99)}$

Where:

S = Standard deviation of the replicate analyses
n = Number of replicates
$t_{(n-1, 1-alpha=0.99)}$ = Student's t value for the 99% confidence level with n-1

For example: t for 8 replicates = $t_{(7,0.99)} = 2.998$

16.2.2 The determined MDL must be less than the concentration spiked but greater than one tenth (1/10) the spiked concentration. If not, repeat the MDL determination at an appropriate spike concentration for affected analytes.

17.0 Pollution Prevention

17.1 Pollution prevention is practiced in the laboratory by minimizing usage of solvents and chemicals, so that disposal of waste generated is held to the smallest amount possible. This is directly linked to the types of extraction procedures in place at the laboratory to reduce the volumes of solvents used for semi-volatile extraction procedures. Northeast Analytical employs extraction procedures such as continuous liquid/liquid and solid phase extraction methods to reduce solvent requirements for water extraction protocols and ASE and Soxhlet extractions for solid matrices.

17.2 Pollution prevention also relies on minimizing to the best extent the chemicals and solvents required to perform extraction and analysis procedures. The laboratory personnel strive to purchase chemicals and standards that will be consumed based on anticipated workload. For additional information about laboratory pollution prevention, please refer to laboratory SOP NE168.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 The GC analyst is responsible for generating the data and also is the initial individual to review the data. This would include inspection of the chromatographic data, processing the raw data, producing all required data forms, inspection of calibration curves for compliance, surrogate recovery, laboratory control spike recovery, matrix spike/matrix spike duplicate recovery, and continuing calibration compliance.

18.2 Once the initial review of the data is performed by the analyst, decisions are made at that time to accept the data if all criteria are met or to reject sample data if any of the quality control parameters or limits are out of control. Depending on the situation, samples requiring re-extraction will be notified to the appropriate extraction personnel, sample extracts requiring re-injection will be queued for analysis, new calibrations may have to be performed, or samples re-analyzed due to failing continuing check standards.

18.3 The analyst may also consult with the quality control officer as to the best form of action to take or if the situation warrants corrective action beyond routine practices. If no recourse is available and the data is to be reported out of criteria, a Case Narrative Report is generated and the deviation is documented and reported to the client. The Case Narrative Report is filed with the data and is also useful for production of case narratives that are issued with the final data reports. If a problem exists that requires follow-up to rectify, a Corrective Action Report (CAR) is issued to document the problem found, steps taken to resolve the problem, and what samples were affected. This CAR form is filed by the quality control officer and reviewed by management to verify that appropriate actions have been taken to correct the problem.

19.0 Corrective Action for Out-Of-Control Data

19.1 The table below outlines the data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

Quality Control Acceptance Criteria and Corrective Action Plan

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	The five point calibration is analyzed initially and when Continuing Calibration Check standard fails criteria.	- %RSD≤20% for the relative response factors for each peak in the five point calibration. - Relative response factors are to be calculated using area for each quantifiable peak with internal standard method.	- Re-analyze the initial calibration standard and/or evaluate/correct instrument malfunction to obtain initial calibration and continuing calibration check standards that meet criteria.
Quality Control Item	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Continuing Calibration Check Standard (CCS)	- Initially analyze a CCS immediately following an initial calibration. - After the initial CCS of the sequence, a CCS must be analyzed after 10 samples. - Analytical sequence must end with analysis of a CCS.	- Calibration factor for the continuing calibration check must be less than 15% difference for all peaks and total PCBs. - Retention time of all quantitated peaks must be within RT window (reset with each initial CCS of a sequence). - The percent recovery for the internal standard (OCN) in the Continuing Calibration Check Standard must be within 50-150% of the OCN area of the associated initial calibration standard. - All samples must be bracketed by a CCS that meet all criteria stated above.	- If the reason for the failure of the CCS appears to be a poor injection (or a degraded standard solution), the CCS will be re-injected (or re-prepared and re-injected) immediately following the failed CCS. This can only occur if the instrument is being attended by an analyst. If upon re-injection, the CCS meets all the acceptance criteria and there is no apparent impact on the sample data (*i.e.*, acceptable OCN areas and surrogate recoveries are observed), the analytical sequence will continue and samples will not be reanalyzed. The associated sample data will be reported. - If CCS failure was not due to a poor injection (or degraded standard solution) or the instrument was unattended at the time of the CCS failure, correct system, if necessary, and recalibrate. Initial calibration and CCS criteria must be met before sample analysis may begin. Samples that are not bracketed by complaint

			CCSs must be reanalyzed. -If acceptable CCSs are observed later in the sequence, samples bracketed by acceptable CCSs will be reported. Samples between the failed CCS and prior/ subsequent complaint CCS will be re-analyzed.
Quality Control Item	**Frequency**	**Acceptance Criteria**	**Corrective Action**
-Retention Time (RT)	- Use the retention time for peak in the CCSs to determine midpoint of the relative retention time window for the analysis sequence. -Each sample analysis: Rely on RT windows to identify PCB congeners to report. Also use pattern recognition and professional judgment for peaks that shift from RT windows, because congener composition may shift RT for GC peaks.	- Each quantitated peak and surrogate peak should be with established windows.	-Inspect chromatographic system for malfunction, correct problem. Perform re-analysis if necessary.
Method Blank	-One per extraction batch of ≤20 samples of the same matrix per day. -Must be analyzed on each instrument used to analyze associated samples. -Must undergo all sample preparative procedures.	- Concentration does not exceed the RL for any PCB congener. - Must meet surrogate criteria of 60-140% recovery.	- Re-analyze method blank to determine if instrument contamination was the cause. If method blank re-analysis passes, then report samples. -If method blank is found to contain PCB contamination above the RL for any PCB congener , then re-extract and re-analyze all associated samples. If no sample exists for re-extraction, report data B flagged to indicate method blank contamination.
Laboratory Control Spike (LCS)	- One per extraction batch of ≤20 samples per matrix per day. The LCS is typically Aroclor 1242.	-Percent recovery of Aroclor 1242 on a total PCB basis must be within method limits of 70-130% -Must meet surrogate criteria of 60-140% recovery.	-Re-analyze LCS to determine if instrument was the cause. If LCS passes, then report samples. -If LCS recovery is still out of limits, the re-extract and re-analyze all associated samples. If no sample exist for re-extraction, report data flagged to indicate LCS failed recovery.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	-Normal method procedure is to extract and analyze a matrix spike sample. One MS per extraction batch of ≤20 samples per matrix per day. The MS is typically Aroclor 1242 -If requested, an MSD can be extracted and analyzed. The MSD would follow the above criteria as for the MS.	- Percent recovery for MS on a total PCB basis should be 70-130%. - If MS/MSD is analyzed, relative percent difference (RPD) should be within 30%. -Must meet surrogate criteria of 60-140% (unless original unspiked sample is also outside of criteria)	-Re-analyze MS and/or MSD to determine if instrument was the cause. If MS and/or MSD pass, then report samples. -Check for errors such as calculations and spike preparation. -Check original unspiked sample results and surrogate recovery for indications of matrix effects. -If no errors are found, and the associated LCS is within 70-130%, then sample matrix effects are likely the cause. Note exceedance in case narrative.
Surrogates	-Surrogates are added to all samples and QC samples. Congener IUPAC 207 (2,2',3,3',4,4',5,6,6'-nonachlorobiphenyl) is used when the sample is prepared for analysis. Decachlorobiphenyl and tetra-chlro-meta-xylene surrogate compounds are used when the sample extract to be analyzed originates from the 8082 analysis.	- Percent recovery for the surrogate should be 60-140%.	-Re-analyze the affected sample or QC sample to determine if instrument was the cause. If surrogate passes, then report samples. -Check for errors in surrogate calculation and surrogate solutions. -If no problem is found, then re-extract and re-analyze the sample. -If re-extraction is within limits and sample extract holding time, then report only the re-analysis. -If the re-extraction is within limits, but out of extraction holding time, then report both sets of data. -If the re-extraction produces surrogate recovery still out of limits, then report both sets of data. -If no sample exist for re-extraction, report data flagged to indicate surrogate failed recovery or have a client re-sample.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Internal Standard	-Octachloronaphthalene (OCN) is added to all sample extracts, QC samples and calibration standards. See text for OCN amounts in calibration standards.	-The internal standard area for samples and QC samples should be 50-150% of the average internal standard area for the associated initial calibration standards. -The internal standard area for CCSs must be 50-150% of the average internal standard area for the initial calibration standards.	-Re-analyze the affected sample or standard to determine if instrument was the cause. If internal standard passes, then report samples. -For standards-If no problem is found, prepare new standards and re-test. If problem still exists, prepare new internal standard and then prepare new standard solution and re-test. -For sample extracts- if no problem is found, then follow procedures outlined above for surrogate corrective action steps for re-extraction and re-analysis.

20.0 Contingencies for Handling Out-Of-Control or Unacceptable Data

20.1 Data that is detected to be out-of-control for any reason, when compared to method acceptance criteria, will addressed in the following manner:

20.1.1 If the problem exists with the gas chromatographic instrumentation, appropriate action will be taken to repair and perform maintenance to bring the instrument back to operation condition. Once the instrumentation is determined to be correctly operating analysis can begin again.

20.1.2 If the problem exists with calibration standard solutions, the analyst will prepare new standards and discard the standard solutions that are suspect. Instrument calibration can be performed and analysis can begin once system is control.

20.1.3 If the problem exists with sample extraction and extract preparation, the extraction step that is producing the out-of-control situation will be diagnosed and rectified. Once the troubleshooting procedures correct the problem extraction can once again occur and analysis can continue.

20.1.4 In situations where data is reported under out-of-control conditions, the data will be annotated with data qualifiers and/or appropriate descriptive comments defining the nature of the excursion in the sample case narrative. If warranted, a corrective action report (CAR) will be issued to define the problem, steps to correct the problem, and final resolution.

21.0 Waste Management

21.1 All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste generated during the execution of this method.

21.2 Please refer to standard operating procedures NE089 and NE054 regarding how hazardous waste is handled and disposed of by the laboratory.

22.0 References:

22.1 U.S. EPA SW-846 "Test Methods for Evaluating Solid waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

22.2 U.S. EPA 40 CFP Part 136, "Guidelines Establishing Test Procedures of the Analysis of Pollutants", July, 1988.

22.3 "Standard Methods for the Examination of Water and Waste Water", 19th Edition 1995, American Public Health Association, American Water Works Association, Water Pollution Control Federation.

22.4 New York State Department of Health, "Environmental Laboratory Approval Program Certification Manual", Wadsworth Center for laboratories and Research, 1996.

22.5 Guide to Environmental Analytical Methods", third edition, Genium Publishing Corporation, 1997.

22.6 Frame, George. M, "A collaborative study of 209 PCB congeners and 6 Aroclors on 20 different HRGC columns 1. Retention and co-elution database.", (1996), Fresenius J Anal. Chem 1141:1-13.

22.7 Frame, George M., " A collaborative study of 209 PCB congeners and 6 Aroclors on 20 different HRGC columns 2. Semi-quantitative Aroclor congener distrbuions." , (1996), Fresenius J Anal. Chem 1142:1-9.

22.8 Frame, George M., Cochran, Jack W., Bowadt, Soren S., "Complete PCB Congener Distributions for Aroclor mixtures determined by 3 HRGC Systems Optimized for Comprehensive, Quantitative, Congener Specific Analysis.", (1996), J. High Resolution Chromatography Vol 19:657-668.

22.9 Frame, George M., Wagner, Robert E., Carnahan, James C., Brown, John F., May, Ralph J., Smullen, Lynn A., Bedard, Donna L., "Comprehensive Quantitative, Congener Specific Analysis of eight Aroclors and Complete PCB Congener assignments on DB-1 Capillary GC Columns" (1996) Chemosphere Vol. 33, No 4 pp.603-623.

23.0 Tables, Diagrams, Flowcharts and Validation Data

Table 1:

Congener IUPAC #	CAS No.	No. of Cl's	Structure unprime-prime	Name
001	2051-60-7	1	2-	Monochlorobiphenyl
002	2051-61-8	1	3-	"
003	2051-62-9	1	4-	"
004	13029-08-8	2	2-2	Dichlorobiphenyl
005	16605-91-7	2	23-	"
006	25569-80-6	2	2-3	"
007	33284-50-3	2	24-	"
008	34883-43-7	2	2-4	"
009	34883-39-1	2	25-	"
010	33146-45-1	2	26-	"
011	2050-67-1	2	3-3	"
012	2974-92-7	2	34-	"
013	2974-90-5	2	3-4	"
014	34883-41-5	2	35-	"
015	2050-68-2	2	4-4	"
016	38444-78-9	3	23-2	Trichlorobiphenyl
017	37680-66-3	3	24-2	"
018	37680-65-2	3	25-2	"
019	38444-73-4	3	26-2	"
020	38444-84-7	3	23-3	"
021	55702-46-0	3	234-	"
022	38444-85-8	3	23-4	"
023	55720-44-0	3	235-	"
024	55702-45-9	3	236-	"
025	55712-37-3	3	24-3	"
026	38444-81-4	3	25-3	"
027	38444-76-7	3	26-3	"
028	7012-37-5	3	24-4	"
029	15862-07-4	3	245-	"
030	35693-92-6	3	246-	"
031	16606-02-3	3	25-4	"
032	38444-77-4	3	26-4	"
033	38444-86-9	3	34-2	"
034	37680-68-5	3	35-2	"
035	37680-69-6	3	34-3	"
036	38444-87-0	3	35-3	"
037	38444-90-5	3	34-4	"
038	53555-66-1	3	345-	"
039	38444-88-1	3	35-4	"
040	38444-93-8	4	23-23	Tetrachlorobiphenyl
041	52663-59-9	4	234-2	"
042	36559-22-5	4	23-24	"
043	70362-46-8	4	235-2	"
044	41464-39-5	4	23-25	"
045	70362-45-7	4	236-2	"
046	41464-47-5	4	23-26	"
047	2437-79-8	4	24-24	"

Congener IUPAC #	CAS No.	No. of Cl's	Structure unprime-prime	Name
048	70362-47-9	4	245-2	"
049	41464-40-8	4	24-25	"
050	62796-65-0	4	246-2	"
051	68194-04-7	4	24-26	"
052	35693-99-3	4	25-25	"
053	41464-41-9	4	25-26	"
054	15968-05-5	4	26-26	"
055	74338-24-2	4	234-3	"
056	41464-43-1	4	23-34	"
057	70424-67-8	4	235-3	"
058	41464-49-7	4	23-35	"
059	74472-33-6	4	236-3	"
060	33025-41-1	4	234-4	"
061	33284-53-6	4	2345-	"
062	54230-22-7	4	2346-	"
063	74472-34-7	4	235-4	"
064	52663-58-8	4	236-4	"
065	33284-54-7	4	2356-	"
066	32598-10-0	4	24-34	"
067	73557-53-8	4	245-3	"
068	73575-52-7	4	24-35	"
069	60233-24-1	4	246-3	"
070	32598-11-1	4	25-34	"
071	41464-46-4	4	26-34	"
072	41464-42-0	4	25-35	"
073	74338-23-1	4	26-35	"
074	32690-93-0	4	245-4	"
075	32598-12-2	4	246-4	"
076	70362-48-0	4	345-2	"
077	32598-13-3	4	34-34	"
078	70362-49-1	4	345-3	"
079	41464-48-6	4	34-35	"
080	33284-52-5	4	35-35	"
081	70362-50-4	4	345-4	"
082	52663-62-4	5	234-23	Pentachlorobiphenyl
083	60145-20-2	5	235-23	"
084	52663-60-2	5	236-23	"
085	65510-45-4	5	234-24	"
086	55312-69-1	5	2345-2	"
087	38380-02-8	5	234-25	"
088	55215-17-3	5	2346-2	"
089	73575-57-2	5	234-26	"
090	68194-07-0	5	235-24	"
091	68194-05-8	5	236-24	"
092	52663-61-3	5	235-25	"
093	73575-56-1	5	2356-2	"
094	73575-55-0	5	235-26	"
095	38379-99-6	5	236-25	"

Congener IUPAC #	CAS No.	No. of Cl's	Structure unprime-prime	Name
096	73575-54-9	5	236-26	"
097	41464-51-1	5	245-23	"
098	60233-25-2	5	246-23	"
099	38380-01-1	5	245-24	"
100	39485-83-1	5	246-24	"
101	37680-73-2	5	245-25	"
102	68194-06-9	5	245-26	"
103	60145-21-3	5	246-25	"
104	56558-16-8	5	246-26	"
105	32598-14-4	5	234-34	"
106	70424-69-0	5	2345-3	"
107	70362-41-3	5	234-35	"
108	74472-35-8	5	2346-3	"
109	70424-68-9	5	235-34	"
110	38380-03-9	5	236-34	"
111	39635-32-0	5	235-35	"
112	74472-36-9	5	2356-3	"
113	68194-10-5	5	236-35	"
114	74472-37-0	5	2345-4	"
115	74472-38-1	5	2346-4	"
116	18259-05-7	5	23456-	"
117	68194-11-6	5	2356-4	"
118	31508-00-6	5	245-34	"
119	56558-17-9	5	246-34	"
120	68194-12-7	5	245-35	"
121	56558-18-0	5	246-35	"
122	76842-07-4	5	345-23	"
123	65510-44-3	5	345-24	"
124	70424-70-3	5	345-25	"
125	74472-39-2	5	345-26	"
126	57465-28-8	5	345-34	"
127	39635-33-1	5	345-35	"
128	38380-07-3	6	234-234	Hexachlorobiphenyl
129	55215-18-4	6	2345-23	"
130	52663-66-8	6	234-235	"
131	61798-70-7	6	2346-23	"
132	38380-05-1	6	234-236	"
133	35694-04-3	6	235-235	"
134	52704-70-8	6	2356-23	"
135	52744-13-5	6	235-236	"
136	38411-22-2	6	236-236	"
137	35694-06-5	6	2345-24	"
138	35065-28-2	6	234-245	"
139	56030-56-9	6	2346-24	"
140	59291-64-4	6	234-246	"
141	52712-04-6	6	2345-25	"
142	41411-61-4	6	23456-2	"
143	68194-15-0	6	2345-26	"
144	68194-14-9	6	2346-25	"

Congener IUPAC #	CAS No.	No. of Cl's	Structure unprime-prime	Name
145	74472-40-5	6	2346-26	"
146	51908-16-8	6	235-245	"
147	68194-13-8	6	2356-24	"
148	74472-41-6	6	235-246	"
149	38380-04-0	6	236-245	"
150	68194-08-1	6	236-246	"
151	52663-63-5	6	2356-25	"
152	68194-09-2	6	2356-26	"
153	35065-27-1	6	245-245	"
154	60145-22-4	6	245-246	"
155	33979-03-2	6	246-246	"
156	38380-08-4	6	2345-34	"
157	69782-90-7	6	234-345	"
158	74472-42-7	6	2346-34	"
159	39635-35-3	6	2345-35	"
160	41411-62-5	6	23456-3	"
161	74474-43-8	6	2346-35	"
162	39635-34-2	6	235-345	"
163	74472-44-9	6	2356-34	"
164	74472-45-0	6	236-345	"
165	74472-46-1	6	2356-35	"
166	41411-63-6	6	23456-4	"
167	52663-72-6	6	245-345	"
168	59291-65-5	6	246-345	"
169	32774-16-6	6	345-345	"
170	35065-30-6	7	2345-234	Heptachlorobiphenyl
171	52663-71-5	7	2346-234	"
172	52663-74-8	7	2345-235	"
173	68194-16-1	7	23456-23	"
174	38411-25-5	7	2345-236	"
175	40186-70-7	7	2346-235	"
176	52663-65-7	7	2346-236	"
177	52663-70-4	7	2356-234	"
178	52663-67-9	7	2356-235	"
179	52663-64-6	7	2356-236	"
180	35065-29-3	7	2345-245	"
181	74472-47-2	7	23456-24	"
182	60145-23-5	7	2345-246	"
183	52663-69-1	7	2346-245	"
184	74472-48-3	7	2346-246	"
185	52712-05-7	7	23456-25	"
186	74472-49-4	7	23456-26	"
187	52663-68-0	7	2356-245	"
188	74487-85-7	7	2356-246	"
189	39635-31-9	7	2345-345	"
190	41411-64-7	7	23456-34	"
191	74472-50-7	7	2346-345	"
192	74472-51-8	7	23456-35	"
193	69782-91-8	7	2356-345	"

Congener IUPAC #	CAS No.	No. of Cl's	Structure unprime-prime	Name
194	35694-08-7	8	2345-2345	Octochlorobiphenyl
195	52663-78-2	8	23456-234	"
196	42740-50-1	8	2345-2346	"
197	33091-17-7	8	2346-2346	"
198	68194-17-2	8	23456-235	"
199	52663-75-9	8	2345-2356	"
200	52663-73-7	8	23456-236	"
201	40186-71-8	8	2346-2356	"
202	2136-99-4	8	2356-2356	"
203	52663-76-0	8	23456-245	"
204	74472-52-9	8	23456-246	"
205	74472-53-0	8	23456-345	"
206	40186-72-9	9	23456-2345	Nonachlorobiphenyl
207	52663-79-3	9	23456-2346	"
208	52663-77-1	9	23456-2356	"
209	2051-24-3	10	23456-23456	Decachlorobiphenyl

Attachment A Solid ASE MDLs

Attachment A

Northeast Analytical Inc.

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Date: 05/25/2005

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: ASE SW-846 Method 3545C
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/1/05

NEA Sample ID	050401SA1	050401SA2	050401SA3	050401SA4	050401SA5	050401SA6	050401SA7	050401SA8	AVERAGE CONCENTRATION	AVERAGE PERCENT RECOVERY	STANDARD DEVIATION	% RELATIVE STANDARD DEVIATION	MDL[2]	Valid[2]
Congener IUPAC#	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	(%)	(ng/g)	(%)	(ng/g)	
1 (1)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	
2 (1)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	
3 (1)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	
4	0.56	0.51	0.54	0.47	0.54	0.62	0.52	0.50	0.53	106.6	0.044	8.19	0.131	Y
5	0.59	0.51	0.60	0.52	0.51	0.52	0.53	0.52	0.54	107.4	0.0359	6.69	0.108	Y
6	0.54	0.53	0.53	0.52	0.53	0.53	0.49	0.52	0.52	104.6	0.0144	2.75	0.043	N
7	0.54	0.54	0.51	0.54	0.52	0.54	0.50	0.54	0.53	105.5	0.0161	3.05	0.048	N
8	0.58	0.49	0.58	0.52	0.52	0.51	0.55	0.55	0.54	107.2	0.0332	6.19	0.100	Y
9	0.53	0.53	0.55	0.53	0.54	0.54	0.48	0.55	0.53	106.1	0.0229	4.32	0.069	Y
10	0.53	0.51	0.55	0.52	0.49	0.53	0.54	0.55	0.53	105.6	0.0203	3.84	0.061	Y
11	0.54	0.49	0.56	0.50	0.51	0.52	0.55	0.50	0.52	103.7	0.0244	4.70	0.073	Y
12	0.57	0.56	0.58	0.51	0.50	0.52	0.52	0.54	0.54	107.5	0.029	5.45	0.088	Y
13	0.57	0.56	0.58	0.51	0.50	0.52	0.52	0.54	0.54	107.5	0.029	5.45	0.088	Y
14	0.55	0.52	0.55	0.52	0.44	0.53	0.49	0.52	0.52	103.1	0.036	7.01	0.108	Y
15	0.53	0.52	0.54	0.49	0.52	0.50	0.53	0.51	0.52	103.5	0.0173	3.33	0.052	Y
16	0.58	0.56	0.62	0.52	0.59	0.57	0.57	0.53	0.57	113.5	0.030	5.22	0.089	Y
17	0.54	0.49	0.56	0.50	0.51	0.52	0.55	0.50	0.52	103.7	0.0244	4.70	0.073	Y
18	0.57	0.56	0.64	0.53	0.51	0.54	0.51	0.52	0.55	109.2	0.042	7.68	0.126	Y
19	0.54	0.50	0.52	0.51	0.53	0.50	0.51	0.51	0.52	103.2	0.0140	2.71	0.042	N
20	0.60	0.52	0.59	0.57	0.53	0.55	0.55	0.51	0.55	110.4	0.0318	5.77	0.095	Y
21	0.54	0.49	0.54	0.54	0.47	0.50	0.52	0.51	0.51	102.9	0.0272	5.29	0.082	Y
22	0.55	0.51	0.55	0.53	0.51	0.53	0.45	0.48	0.51	102.4	0.033	6.52	0.100	Y
23	0.53	0.50	0.57	0.51	0.50	0.53	0.49	0.50	0.52	103.2	0.0280	5.42	0.084	Y
24	0.57	0.56	0.58	0.51	0.50	0.52	0.52	0.54	0.54	107.5	0.029	5.45	0.088	Y
25	0.57	0.54	0.54	0.52	0.50	0.52	0.50	0.52	0.53	105.1	0.024	4.52	0.071	Y
26	0.56	0.51	0.54	0.53	0.53	0.54	0.51	0.54	0.53	106.0	0.017	3.21	0.051	Y
27	0.55	0.53	0.52	0.54	0.48	0.52	0.49	0.51	0.52	103.5	0.0220	4.26	0.066	Y
28	0.60	0.52	0.59	0.57	0.53	0.55	0.55	0.51	0.55	110.4	0.0318	5.77	0.095	Y
29	0.56	0.51	0.54	0.53	0.53	0.54	0.51	0.54	0.53	106.0	0.017	3.21	0.051	Y
30	0.54	0.51	0.56	0.52	0.48	0.53	0.55	0.51	0.52	104.8	0.0261	4.98	0.078	Y
31	0.55	0.50	0.54	0.56	0.52	0.54	0.49	0.49	0.52	104.8	0.0281	5.35	0.084	Y
32	0.54	0.51	0.55	0.53	0.49	0.51	0.51	0.49	0.52	103.1	0.023	4.45	0.069	Y

Attachment A Solid ASE MDLs

Attachment A

Northeast Analytical Inc.

Date: 05/25/2005

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: ASE SW-846 Method 3545C
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/1/05

33	0.60	0.50	0.58	0.57	0.53	0.54	0.50	0.50	0.54	108.0	0.039	7.14	0.116	Y
34	0.52	0.52	0.59	0.58	0.48	0.55	0.52	0.49	0.53	105.9	0.0381	7.19	0.114	Y
35	0.57	0.56	0.56	0.55	0.51	0.51	0.54	0.54	0.54	108.3	0.0220	4.06	0.066	Y
36	0.53	0.54	0.54	0.54	0.51	0.54	0.53	0.53	0.53	106.9	0.0101	1.90	0.030	N
37	0.50	0.48	0.47	0.55	0.44	0.49	0.50	0.48	0.49	97.7	0.0309	6.32	0.093	Y
38	0.58	0.52	0.62	0.54	0.52	0.55	0.52	0.50	0.54	108.6	0.0367	6.75	0.110	Y
39	0.57	0.52	0.55	0.54	0.53	0.54	0.52	0.51	0.53	106.8	0.0187	3.50	0.056	Y
40	0.57	0.56	0.56	0.55	0.51	0.51	0.54	0.54	0.54	108.3	0.0220	4.06	0.066	Y
41	0.53	0.51	0.54	0.52	0.50	0.54	0.55	0.56	0.53	106.0	0.0192	3.63	0.058	Y
42	0.58	0.52	0.62	0.54	0.52	0.55	0.52	0.50	0.54	108.6	0.0367	6.75	0.110	Y
43	0.52	0.51	0.56	0.53	0.51	0.51	0.48	0.54	0.52	103.7	0.0232	4.47	0.069	Y
44	0.57	0.52	0.55	0.54	0.53	0.54	0.52	0.51	0.53	106.8	0.0187	3.50	0.056	Y
45	0.60	0.52	0.59	0.57	0.53	0.55	0.55	0.51	0.55	110.4	0.0318	5.77	0.095	Y
46	0.55	0.51	0.49	0.53	0.51	0.54	0.53	0.50	0.52	103.8	0.0194	3.74	0.058	Y
47	0.57	0.52	0.55	0.54	0.53	0.54	0.52	0.51	0.53	106.8	0.0187	3.50	0.056	Y
48	0.52	0.57	0.51	0.54	0.51	0.52	0.54	0.49	0.52	104.8	0.0237	4.52	0.071	Y
49	0.53	0.54	0.54	0.54	0.51	0.54	0.53	0.53	0.53	106.9	0.0101	1.90	0.030	N
50	0.58	0.53	0.57	0.52	0.48	0.54	0.50	0.55	0.53	106.7	0.0323	6.05	0.097	Y
51	0.60	0.52	0.59	0.57	0.53	0.55	0.55	0.51	0.55	110.4	0.0318	5.77	0.095	Y
52	0.55	0.50	0.55	0.54	0.53	0.55	0.52	0.54	0.53	106.7	0.019	3.64	0.058	Y
53	0.57	0.54	0.54	0.52	0.50	0.52	0.50	0.52	0.53	105.1	0.024	4.52	0.071	Y
54	0.60	0.47	0.59	0.53	0.50	0.52	0.50	0.53	0.53	105.8	0.0429	8.12	0.129	Y
55	0.55	0.52	0.53	0.54	0.51	0.53	0.51	0.51	0.52	105.0	0.0168	3.20	0.050	Y
56	0.55	0.49	0.53	0.51	0.52	0.49	0.47	0.53	0.51	102.0	0.0257	5.04	0.077	Y
57	0.54	0.50	0.55	0.52	0.50	0.51	0.55	0.52	0.52	104.9	0.0215	4.10	0.065	Y
58	0.55	0.52	0.54	0.55	0.52	0.53	0.51	0.53	0.53	106.3	0.0156	2.94	0.047	N
59	0.57	0.57	0.56	0.57	0.51	0.56	0.56	0.53	0.55	110.6	0.0208	3.75	0.062	Y
60	0.55	0.55	0.55	0.52	0.54	0.54	0.50	0.52	0.53	106.3	0.0186	3.50	0.056	Y
61	0.56	0.51	0.55	0.55	0.49	0.52	0.53	0.51	0.53	105.6	0.0257	4.87	0.077	Y
62	0.53	0.53	0.55	0.53	0.51	0.54	0.50	0.52	0.52	104.9	0.0159	3.03	0.048	N
63	0.56	0.54	0.55	0.55	0.50	0.52	0.54	0.52	0.54	107.1	0.0200	3.74	0.060	Y
64	0.52	0.50	0.50	0.54	0.47	0.50	0.50	0.52	0.51	101.4	0.0213	4.21	0.064	Y

Attachment A Solid ASE MDLs

Attachment A

Northeast Analytical Inc.

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Date: 05/25/2005

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: ASE SW-846 Method 3545C
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/1/05

65	0.57	0.52	0.55	0.54	0.53	0.54	0.52	0.51	0.53	106.8	0.0187	3.50	0.056	Y
66	0.55	0.52	0.55	0.53	0.49	0.52	0.50	0.52	0.52	104.6	0.0218	4.16	0.065	Y
67	0.55	0.52	0.52	0.55	0.52	0.52	0.50	0.52	0.52	104.9	0.0174	3.31	0.052	Y
68	0.54	0.53	0.50	0.55	0.49	0.54	0.53	0.52	0.52	104.7	0.0206	3.93	0.062	Y
69	0.54	0.53	0.54	0.57	0.49	0.53	0.52	0.51	0.53	105.7	0.0217	4.11	0.065	Y
70	0.59	0.53	0.63	0.54	0.50	0.52	0.52	0.50	0.54	108.6	0.0460	8.46	0.138	Y
71	0.57	0.56	0.56	0.55	0.51	0.51	0.54	0.54	0.54	108.3	0.0220	4.06	0.066	Y
72	0.54	0.51	0.56	0.54	0.50	0.54	0.51	0.51	0.53	105.4	0.0227	4.31	0.068	Y
73	0.55	0.51	0.54	0.53	0.50	0.54	0.51	0.54	0.53	105.6	0.018	3.46	0.055	Y
74	0.59	0.53	0.63	0.54	0.50	0.52	0.52	0.50	0.54	108.6	0.0460	8.46	0.138	Y
75	0.53	0.53	0.55	0.53	0.51	0.54	0.50	0.52	0.52	104.9	0.0159	3.03	0.048	N
76	0.48	0.54	0.51	0.53	0.48	0.52	0.53	0.52	0.51	102.9	0.021	4.09	0.063	Y
77	0.56	0.55	0.54	0.55	0.53	0.54	0.51	0.55	0.54	108.4	0.0147	2.71	0.044	N
78	0.51	0.47	0.56	0.52	0.49	0.52	0.50	0.51	0.51	101.8	0.0264	5.18	0.079	Y
79	0.54	0.46	0.48	0.54	0.48	0.43	0.51	0.50	0.49	98.3	0.0385	7.83	0.115	Y
80	0.55	0.54	0.53	0.53	0.53	0.55	0.52	0.50	0.53	106.2	0.0159	3.00	0.048	N
81	0.51	0.52	0.53	0.52	0.45	0.49	0.50	0.50	0.50	100.6	0.025	5.00	0.075	Y
82	0.57	0.53	0.57	0.55	0.52	0.55	0.53	0.52	0.54	108.2	0.0189	3.50	0.057	Y
83	0.55	0.55	0.53	0.54	0.53	0.54	0.53	0.53	0.54	107.4	0.0097	1.80	0.029	N
84	0.52	0.53	0.56	0.57	0.53	0.51	0.50	0.52	0.53	106.0	0.0255	4.81	0.076	Y
85	0.56	0.50	0.55	0.54	0.51	0.54	0.50	0.51	0.53	105.1	0.0233	4.44	0.070	Y
86	0.58	0.53	0.58	0.53	0.53	0.55	0.55	0.51	0.55	109.0	0.0238	4.37	0.071	Y
87	0.52	0.52	0.50	0.50	0.46	0.55	0.53	0.55	0.52	103.2	0.0285	5.52	0.085	Y
88	0.56	0.54	0.55	0.55	0.50	0.52	0.54	0.52	0.54	107.1	0.0200	3.74	0.060	Y
89	0.55	0.52	0.53	0.54	0.51	0.53	0.51	0.51	0.52	105.0	0.0168	3.20	0.050	Y
90	0.56	0.54	0.57	0.53	0.52	0.54	0.56	0.53	0.54	108.7	0.0175	3.22	0.053	Y
91	0.56	0.51	0.55	0.55	0.49	0.52	0.53	0.51	0.53	105.6	0.0257	4.87	0.077	Y
92	0.55	0.54	0.53	0.53	0.53	0.55	0.52	0.50	0.53	106.2	0.0159	3.00	0.048	N
93	0.55	0.52	0.54	0.55	0.52	0.53	0.51	0.53	0.53	106.3	0.0156	2.94	0.047	N
94	0.54	0.53	0.50	0.55	0.49	0.54	0.53	0.52	0.52	104.7	0.0206	3.93	0.062	Y
95	0.57	0.53	0.57	0.55	0.52	0.53	0.50	0.54	0.54	107.6	0.0260	4.84	0.078	Y
96	0.57	0.57	0.56	0.57	0.51	0.56	0.56	0.53	0.55	110.6	0.0208	3.75	0.062	Y

Attachment A Solid ASE MDLs

Attachment A

Northeast Analytical Inc.

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Date: 05/25/2005

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: ASE SW-846 Method 3545C
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/1/05

97	0.58	0.53	0.58	0.53	0.53	0.55	0.55	0.51	0.55	109.0	0.0238	4.37	0.071	Y
98	0.55	0.52	0.52	0.55	0.52	0.52	0.50	0.52	0.52	104.9	0.0174	3.31	0.052	Y
99	0.53	0.53	0.61	0.54	0.53	0.53	0.52	0.54	0.54	108.4	0.0291	5.36	0.087	Y
100	0.57	0.53	0.57	0.55	0.52	0.53	0.50	0.54	0.54	107.6	0.0260	4.84	0.078	Y
101	0.56	0.54	0.57	0.53	0.52	0.54	0.56	0.53	0.54	108.7	0.0175	3.22	0.053	Y
102	0.55	0.52	0.54	0.55	0.52	0.53	0.51	0.53	0.53	106.3	0.0156	2.94	0.047	N
103	0.54	0.51	0.56	0.54	0.50	0.54	0.51	0.51	0.53	105.4	0.0227	4.31	0.068	Y
104	0.53	0.57	0.53	0.55	0.52	0.55	0.55	0.50	0.54	107.6	0.0224	4.15	0.067	Y
105	0.55	0.52	0.56	0.54	0.50	0.52	0.51	0.49	0.52	104.5	0.0253	4.84	0.076	Y
106	0.56	0.49	0.52	0.52	0.51	0.53	0.52	0.52	0.52	104.5	0.0203	3.89	0.061	Y
107	0.54	0.54	0.54	0.54	0.52	0.53	0.53	0.52	0.53	106.4	0.0085	1.60	0.025	N
108	0.58	0.53	0.58	0.53	0.53	0.55	0.55	0.51	0.55	109.0	0.0238	4.37	0.071	Y
109	0.54	0.54	0.54	0.54	0.52	0.53	0.53	0.52	0.53	106.4	0.0085	1.60	0.025	N
110	0.56	0.53	0.59	0.53	0.50	0.54	0.55	0.52	0.54	108.1	0.0258	4.78	0.077	Y
111	0.56	0.55	0.54	0.55	0.53	0.54	0.51	0.55	0.54	108.4	0.0147	2.71	0.044	N
112	0.49	0.51	0.52	0.51	0.45	0.49	0.54	0.50	0.50	100.4	0.028	5.52	0.083	Y
113	0.56	0.54	0.57	0.53	0.52	0.54	0.56	0.53	0.54	108.7	0.0175	3.22	0.053	Y
114	0.57	0.50	0.48	0.52	0.51	0.53	0.51	0.52	0.52	103.2	0.0262	5.07	0.078	Y
115	0.56	0.53	0.59	0.53	0.50	0.54	0.55	0.52	0.54	108.1	0.0258	4.78	0.077	Y
116	0.56	0.50	0.55	0.54	0.51	0.54	0.50	0.51	0.53	105.1	0.0233	4.44	0.070	Y
117	0.56	0.50	0.55	0.54	0.51	0.54	0.50	0.51	0.53	105.1	0.0233	4.44	0.070	Y
118	0.59	0.49	0.56	0.54	0.53	0.59	0.52	0.51	0.54	108.2	0.0353	6.53	0.106	Y
119	0.58	0.53	0.58	0.53	0.53	0.55	0.55	0.51	0.55	109.0	0.0238	4.37	0.071	Y
120	0.57	0.50	0.52	0.53	0.49	0.50	0.50	0.51	0.52	103.3	0.025	4.93	0.076	Y
121	0.53	0.53	0.51	0.54	0.49	0.53	0.51	0.53	0.52	104.6	0.0172	3.29	0.052	Y
122	0.55	0.51	0.54	0.51	0.52	0.54	0.49	0.51	0.52	104.3	0.0206	3.94	0.062	Y
123	0.60	0.56	0.55	0.51	0.52	0.58	0.51	0.52	0.54	108.3	0.0356	6.57	0.107	Y
124	0.54	0.54	0.54	0.54	0.52	0.53	0.53	0.52	0.53	106.4	0.0085	1.60	0.025	N
125	0.58	0.57	0.57	0.54	0.52	0.57	0.55	0.48	0.55	109.6	0.0326	5.95	0.098	Y
126	0.55	0.56	0.54	0.54	0.50	0.55	0.50	0.50	0.53	106.2	0.0238	4.48	0.071	Y
127	0.52	0.52	0.59	0.55	0.52	0.53	0.53	0.52	0.53	106.8	0.0237	4.43	0.071	Y
128	0.57	0.55	0.54	0.54	0.52	0.55	0.53	0.53	0.54	108.2	0.017	3.15	0.051	Y

Attachment A Solid ASE MDLs

Attachment A

Northeast Analytical Inc.

Date: 05/25/2005

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: ASE SW-846 Method 3545C
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/1/05

129	0.55	0.51	0.58	0.54	0.52	0.55	0.52	0.53	0.54	107.7	0.0211	3.91	0.063	Y
130	0.52	0.48	0.53	0.50	0.52	0.50	0.54	0.53	0.52	103.3	0.019	3.59	0.056	Y
131	0.53	0.51	0.53	0.53	0.51	0.53	0.52	0.52	0.52	104.6	0.0098	1.87	0.029	N
132	0.60	0.51	0.57	0.51	0.56	0.60	0.55	0.53	0.55	110.7	0.0350	6.32	0.105	Y
133	0.60	0.52	0.54	0.53	0.49	0.52	0.49	0.51	0.52	104.8	0.0345	6.59	0.103	Y
134	0.58	0.55	0.56	0.53	0.49	0.54	0.52	0.51	0.53	106.9	0.0295	5.52	0.088	Y
135	0.52	0.54	0.54	0.57	0.52	0.56	0.54	0.52	0.54	107.6	0.0203	3.78	0.061	Y
136	0.55	0.55	0.53	0.54	0.53	0.54	0.53	0.53	0.54	107.4	0.0097	1.80	0.029	N
137	0.55	0.48	0.52	0.49	0.49	0.50	0.54	0.53	0.51	102.5	0.0256	5.00	0.077	Y
138	0.55	0.51	0.58	0.54	0.52	0.55	0.52	0.53	0.54	107.7	0.0211	3.91	0.063	Y
139	0.57	0.55	0.53	0.54	0.48	0.51	0.50	0.51	0.52	104.6	0.0278	5.32	0.083	Y
140	0.57	0.55	0.53	0.54	0.48	0.51	0.50	0.51	0.52	104.6	0.0278	5.32	0.083	Y
141	0.54	0.52	0.54	0.53	0.51	0.53	0.51	0.50	0.52	104.1	0.0138	2.66	0.041	N
142	0.53	0.51	0.53	0.53	0.51	0.53	0.52	0.52	0.52	104.6	0.0098	1.87	0.029	N
143	0.58	0.55	0.56	0.53	0.49	0.54	0.52	0.51	0.53	106.9	0.0295	5.52	0.088	Y
144	0.56	0.52	0.52	0.56	0.51	0.53	0.52	0.52	0.53	106.0	0.0195	3.68	0.058	Y
145	0.54	0.55	0.55	0.56	0.55	0.54	0.51	0.52	0.54	108.1	0.0191	3.53	0.057	Y
146	0.55	0.52	0.56	0.54	0.50	0.52	0.51	0.49	0.52	104.5	0.0253	4.84	0.076	Y
147	0.58	0.54	0.58	0.55	0.51	0.53	0.53	0.52	0.54	108.2	0.0260	4.80	0.078	Y
148	0.57	0.53	0.57	0.55	0.52	0.55	0.53	0.52	0.54	108.2	0.0189	3.50	0.057	Y
149	0.58	0.54	0.58	0.55	0.51	0.53	0.53	0.52	0.54	108.2	0.0260	4.80	0.078	Y
150	0.56	0.54	0.57	0.53	0.52	0.54	0.56	0.53	0.54	108.7	0.0175	3.22	0.053	Y
151	0.56	0.55	0.54	0.55	0.53	0.54	0.51	0.55	0.54	108.4	0.0147	2.71	0.044	N
152	0.53	0.51	0.52	0.51	0.47	0.54	0.51	0.54	0.52	103.2	0.0243	4.71	0.073	Y
153	0.56	0.55	0.57	0.54	0.52	0.54	0.51	0.52	0.54	107.9	0.0211	3.91	0.063	Y
154	0.52	0.54	0.54	0.57	0.52	0.56	0.54	0.52	0.54	107.6	0.0203	3.78	0.061	Y
155	0.53	0.51	0.52	0.51	0.47	0.54	0.51	0.54	0.52	103.2	0.0243	4.71	0.073	Y
156	0.53	0.52	0.57	0.53	0.52	0.54	0.51	0.53	0.53	106.3	0.0185	3.48	0.055	Y
157	0.53	0.52	0.57	0.53	0.52	0.54	0.51	0.53	0.53	106.3	0.0185	3.48	0.055	Y
158	0.55	0.52	0.59	0.55	0.50	0.54	0.52	0.54	0.54	107.9	0.0283	5.24	0.085	Y
159	0.57	0.50	0.51	0.52	0.46	0.51	0.52	0.51	0.51	102.8	0.0297	5.78	0.089	Y
160	0.51	0.45	0.54	0.51	0.47	0.51	0.52	0.53	0.50	101.0	0.0288	5.70	0.086	Y

Attachment A Solid ASE MDLs

Attachment A

Northeast Analytical Inc.

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Date: 05/25/2005

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: ASE SW-846 Method 3545C
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/1/05

161	0.50	0.53	0.53	0.51	0.50	0.52	0.50	0.49	0.51	102.4	0.0148	2.89	0.044	N
162	0.56	0.57	0.57	0.52	0.51	0.55	0.55	0.53	0.54	108.9	0.0227	4.17	0.068	Y
163	0.55	0.51	0.58	0.54	0.52	0.55	0.52	0.53	0.54	107.7	0.0211	3.91	0.063	Y
164	0.51	0.48	0.57	0.52	0.47	0.52	0.53	0.54	0.52	103.2	0.0335	6.49	0.100	Y
165	0.56	0.54	0.56	0.56	0.50	0.54	0.52	0.50	0.53	106.9	0.0241	4.51	0.072	Y
166	0.57	0.55	0.54	0.54	0.52	0.55	0.53	0.53	0.54	108.2	0.017	3.15	0.051	Y
167	0.49	0.50	0.58	0.53	0.54	0.54	0.52	0.55	0.53	106.3	0.0303	5.69	0.091	Y
168	0.56	0.55	0.57	0.54	0.52	0.54	0.51	0.52	0.54	107.9	0.0211	3.91	0.063	Y
169	0.52	0.52	0.51	0.49	0.48	0.49	0.48	0.53	0.50	100.3	0.0196	3.91	0.059	Y
170	0.52	0.50	0.56	0.53	0.51	0.50	0.51	0.54	0.52	104.3	0.0189	3.62	0.057	Y
171	0.54	0.54	0.57	0.52	0.52	0.54	0.53	0.49	0.53	106.0	0.0226	4.27	0.068	Y
172	0.52	0.50	0.52	0.53	0.40	0.49	0.54	0.53	0.50	100.6	0.0451	8.97	0.135	Y
173	0.54	0.54	0.57	0.52	0.52	0.54	0.53	0.49	0.53	106.0	0.0226	4.27	0.068	Y
174	0.54	0.53	0.55	0.53	0.55	0.52	0.54	0.50	0.53	106.6	0.0156	2.93	0.047	N
175	0.52	0.48	0.54	0.52	0.42	0.50	0.52	0.51	0.50	100.5	0.0374	7.45	0.112	Y
176	0.51	0.53	0.50	0.53	0.49	0.51	0.50	0.52	0.51	102.1	0.0146	2.85	0.044	N
177	0.53	0.53	0.53	0.50	0.53	0.51	0.50	0.50	0.52	103.1	0.0155	3.01	0.047	N
178	0.52	0.46	0.55	0.56	0.46	0.57	0.50	0.50	0.52	103.1	0.042	8.15	0.126	Y
179	0.59	0.51	0.53	0.56	0.50	0.51	0.50	0.52	0.53	105.3	0.032	6.03	0.095	Y
180	0.56	0.51	0.54	0.52	0.51	0.54	0.53	0.51	0.53	105.4	0.0195	3.71	0.059	Y
181	0.52	0.51	0.56	0.52	0.49	0.51	0.52	0.51	0.52	103.3	0.0198	3.82	0.059	Y
182	0.55	0.56	0.54	0.54	0.50	0.55	0.50	0.50	0.53	106.2	0.0238	4.48	0.071	Y
183	0.54	0.49	0.52	0.50	0.38	0.50	0.51	0.49	0.49	98.4	0.0486	9.87	0.146	Y
184	0.56	0.54	0.56	0.56	0.50	0.54	0.52	0.50	0.53	106.9	0.0241	4.51	0.072	Y
185	0.52	0.53	0.54	0.53	0.46	0.53	0.51	0.52	0.52	103.3	0.0245	4.75	0.074	Y
186	0.62	0.56	0.60	0.54	0.52	0.54	0.51	0.51	0.55	109.6	0.0406	7.42	0.122	Y
187	0.52	0.53	0.53	0.53	0.49	0.51	0.52	0.51	0.52	103.4	0.0134	2.60	0.040	N
188	0.58	0.49	0.55	0.50	0.53	0.58	0.49	0.52	0.53	106.1	0.0363	6.84	0.109	Y
189	0.55	0.55	0.54	0.54	0.59	0.55	0.54	0.53	0.55	109.6	0.0174	3.18	0.052	Y
190	0.55	0.49	0.58	0.53	0.48	0.50	0.52	0.51	0.52	104.0	0.0322	6.19	0.097	Y
191	0.54	0.53	0.54	0.54	0.49	0.51	0.51	0.53	0.52	104.7	0.0181	3.46	0.054	Y
192	0.55	0.50	0.55	0.52	0.52	0.54	0.51	0.53	0.53	105.7	0.0196	3.71	0.059	Y

Attachment A Solid ASE MDLs

Attachment A

Northeast Analytical Inc.

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Date: 05/25/2005

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: ASE SW-846 Method 3545C
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/1/05

193	0.56	0.51	0.54	0.52	0.51	0.54	0.53	0.51	0.53	105.4	0.0195	3.71	0.059	Y
194	0.56	0.50	0.54	0.52	0.45	0.50	0.49	0.52	0.51	102.3	0.0339	6.63	0.102	Y
195	0.54	0.52	0.52	0.51	0.51	0.52	0.48	0.51	0.51	102.6	0.0173	3.37	0.052	Y
196	0.54	0.53	0.54	0.56	0.52	0.54	0.50	0.50	0.53	105.8	0.0197	3.72	0.059	Y
197	0.53	0.52	0.57	0.53	0.52	0.54	0.51	0.53	0.53	106.3	0.0185	3.48	0.055	Y
198	0.58	0.54	0.59	0.55	0.52	0.53	0.51	0.52	0.54	108.6	0.0278	5.11	0.083	Y
199	0.58	0.54	0.59	0.55	0.52	0.53	0.51	0.52	0.54	108.6	0.0278	5.11	0.083	Y
200	0.52	0.52	0.54	0.51	0.45	0.53	0.50	0.52	0.51	102.3	0.0293	5.73	0.088	Y
201	0.54	0.50	0.53	0.55	0.46	0.55	0.53	0.48	0.52	103.6	0.0333	6.44	0.100	Y
202	0.56	0.57	0.57	0.52	0.51	0.55	0.55	0.53	0.54	108.9	0.0227	4.17	0.068	Y
203	0.55	0.53	0.52	0.53	0.45	0.52	0.50	0.50	0.51	102.4	0.029	5.65	0.087	Y
204	0.56	0.50	0.55	0.55	0.46	0.51	0.48	0.49	0.51	102.6	0.0353	6.89	0.106	Y
205	0.56	0.54	0.56	0.53	0.47	0.53	0.55	0.53	0.53	106.7	0.0276	5.18	0.083	Y
206	0.53	0.49	0.54	0.53	0.44	0.53	0.51	0.51	0.51	102.0	0.032	6.18	0.095	Y
207	0.55	0.55	0.54	0.54	0.59	0.55	0.54	0.53	0.55	109.6	0.0174	3.18	0.052	Y
208	0.54	0.56	0.56	0.52	0.70	0.53	0.53	0.52	0.56	111.5	0.0617	11.07	0.185	Y
209	0.54	0.54	0.54	0.54	0.45	0.51	0.50	0.51	0.51	102.9	0.0320	6.21	0.096	Y

Attachment A Solid SOX MDLs

Attachment A

Northeast Analytical Inc.

Date: 05/25/2005

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: Soxhlet
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/5/05

NEA Sample ID	050405SS1RR'	050405SS2RR'	050405SS3	050405SS4	050405SS5	050405SS6RR'	050405SS7	050405SS8RR1	AVERAGE CONCENTRATION	AVERAGE PERCENT RECOVERY	STANDARD DEVIATION	% RELATIVE STANDARD DEVIATION	MDL[2]	Valid[2]
Congener IUPAC#	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	(%)	(ng/g)	(%)	(ng/g)	
1 (1)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	
2 (1)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	
3 (1)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	
4	0.54	0.56	0.48	0.55	0.54	0.54	0.56	0.52	0.53	106.8	0.027	5.01	0.080	Y
5	0.58	0.53	0.53	0.50	0.54	0.50	0.51	0.52	0.53	105.4	0.0267	5.06	0.080	Y
6	0.58	0.54	0.51	0.56	0.54	0.54	0.58	0.52	0.54	108.7	0.0257	4.73	0.077	Y
7	0.55	0.54	0.50	0.53	0.49	0.51	0.56	0.52	0.53	105.1	0.0253	4.81	0.076	Y
8	0.58	0.54	0.51	0.52	0.52	0.50	0.53	0.51	0.53	105.3	0.0265	5.03	0.079	Y
9	0.57	0.51	0.50	0.51	0.50	0.50	0.55	0.58	0.53	105.8	0.0347	6.57	0.104	Y
10	0.54	0.58	0.51	0.53	0.50	0.48	0.56	0.53	0.53	105.7	0.0303	5.74	0.091	Y
11	0.55	0.55	0.49	0.51	0.49	0.50	0.56	0.56	0.53	105.1	0.0308	5.86	0.092	Y
12	0.51	0.53	0.50	0.54	0.54	0.51	0.54	0.56	0.53	105.9	0.019	3.58	0.057	Y
13	0.51	0.53	0.50	0.54	0.54	0.51	0.54	0.56	0.53	105.9	0.019	3.58	0.057	Y
14	0.54	0.55	0.54	0.54	0.56	0.54	0.53	0.58	0.55	109.5	0.014	2.53	0.041	N
15	0.56	0.51	0.53	0.51	0.51	0.50	0.58	0.54	0.53	105.7	0.0272	5.14	0.081	Y
16	0.57	0.55	0.52	0.51	0.55	0.56	0.52	0.59	0.55	109.4	0.030	5.42	0.089	Y
17	0.55	0.55	0.49	0.51	0.49	0.50	0.56	0.56	0.53	105.1	0.0308	5.86	0.092	Y
18	0.51	0.53	0.54	0.53	0.56	0.49	0.56	0.55	0.53	106.5	0.024	4.56	0.073	Y
19	0.57	0.54	0.50	0.54	0.51	0.50	0.56	0.53	0.53	106.3	0.0261	4.91	0.078	Y
20	0.55	0.53	0.56	0.54	0.55	0.54	0.55	0.58	0.55	109.9	0.0139	2.52	0.042	N
21	0.58	0.54	0.52	0.54	0.54	0.56	0.56	0.56	0.55	110.2	0.0172	3.13	0.052	Y
22	0.53	0.57	0.52	0.57	0.56	0.54	0.54	0.55	0.55	109.6	0.019	3.43	0.056	Y
23	0.54	0.56	0.53	0.52	0.55	0.52	0.58	0.58	0.55	109.5	0.0234	4.28	0.070	Y
24	0.51	0.53	0.50	0.54	0.54	0.51	0.54	0.56	0.53	105.9	0.019	3.58	0.057	Y
25	0.55	0.51	0.54	0.53	0.50	0.52	0.53	0.54	0.53	105.7	0.016	3.00	0.048	N
26	0.54	0.55	0.56	0.56	0.57	0.54	0.59	0.55	0.56	111.4	0.016	2.79	0.047	N
27	0.52	0.52	0.52	0.54	0.52	0.54	0.55	0.56	0.54	107.2	0.0152	2.83	0.045	N
28	0.55	0.53	0.56	0.54	0.55	0.54	0.55	0.58	0.55	109.9	0.0139	2.52	0.042	N
29	0.54	0.55	0.56	0.56	0.57	0.54	0.59	0.55	0.56	111.4	0.016	2.79	0.047	N
30	0.57	0.52	0.51	0.50	0.53	0.51	0.55	0.53	0.53	105.5	0.0246	4.66	0.074	Y
31	0.54	0.51	0.54	0.54	0.55	0.52	0.55	0.59	0.54	108.4	0.0244	4.49	0.073	Y
32	0.54	0.50	0.49	0.53	0.53	0.49	0.54	0.53	0.52	103.6	0.021	4.09	0.064	Y

Attachment A Solid SOX MDLs

Attachment A

Northeast Analytical Inc.
Quality Assurance/Quality Control
PCB Congener Specific Method Detection Limit Study

Date: 05/25/2005

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: Soxhlet
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/5/05

33	0.55	0.56	0.51	0.55	0.57	0.53	0.55	0.59	0.55	110.1	0.024	4.37	0.072	Y
34	0.56	0.54	0.53	0.54	0.53	0.54	0.59	0.56	0.55	109.9	0.0189	3.44	0.057	Y
35	0.54	0.54	0.51	0.53	0.56	0.52	0.59	0.58	0.55	109.3	0.0276	5.05	0.083	Y
36	0.57	0.54	0.53	0.55	0.52	0.54	0.52	0.55	0.54	108.1	0.0153	2.84	0.046	N
37	0.55	0.52	0.52	0.52	0.55	0.56	0.56	0.52	0.54	107.0	0.0193	3.60	0.058	Y
38	0.53	0.53	0.53	0.55	0.56	0.51	0.56	0.58	0.54	108.7	0.0223	4.10	0.067	Y
39	0.56	0.55	0.52	0.53	0.53	0.56	0.55	0.55	0.54	108.4	0.0133	2.45	0.040	N
40	0.54	0.54	0.51	0.53	0.56	0.52	0.59	0.58	0.55	109.3	0.0276	5.05	0.083	Y
41	0.51	0.57	0.51	0.51	0.50	0.51	0.55	0.56	0.53	105.7	0.0280	5.29	0.084	Y
42	0.53	0.53	0.53	0.55	0.56	0.51	0.56	0.58	0.54	108.7	0.0223	4.10	0.067	Y
43	0.57	0.52	0.48	0.53	0.53	0.52	0.55	0.59	0.54	107.3	0.0334	6.23	0.100	Y
44	0.56	0.55	0.52	0.53	0.53	0.56	0.55	0.55	0.54	108.4	0.0133	2.45	0.040	N
45	0.55	0.53	0.56	0.54	0.55	0.54	0.55	0.58	0.55	109.9	0.0139	2.52	0.042	N
46	0.55	0.54	0.54	0.56	0.52	0.51	0.54	0.54	0.54	107.7	0.0168	3.12	0.050	Y
47	0.56	0.55	0.52	0.53	0.53	0.56	0.55	0.55	0.54	108.4	0.0133	2.45	0.040	N
48	0.54	0.51	0.52	0.57	0.52	0.53	0.53	0.52	0.53	106.0	0.0178	3.36	0.053	Y
49	0.57	0.54	0.53	0.55	0.52	0.54	0.52	0.55	0.54	108.1	0.0153	2.84	0.046	N
50	0.55	0.54	0.50	0.57	0.54	0.51	0.58	0.55	0.54	108.4	0.0282	5.21	0.085	Y
51	0.55	0.53	0.56	0.54	0.55	0.54	0.55	0.58	0.55	109.9	0.0139	2.52	0.042	N
52	0.55	0.55	0.51	0.54	0.52	0.51	0.56	0.61	0.54	108.7	0.035	6.39	0.104	Y
53	0.55	0.51	0.54	0.53	0.50	0.52	0.53	0.54	0.53	105.7	0.016	3.00	0.048	N
54	0.57	0.52	0.50	0.56	0.53	0.56	0.54	0.53	0.54	107.7	0.0228	4.24	0.068	Y
55	0.53	0.55	0.48	0.54	0.53	0.53	0.57	0.57	0.54	107.8	0.0276	5.12	0.083	Y
56	0.54	0.53	0.48	0.53	0.50	0.53	0.55	0.52	0.52	104.8	0.0243	4.65	0.073	Y
57	0.57	0.55	0.51	0.57	0.53	0.53	0.57	0.51	0.54	108.8	0.0264	4.85	0.079	Y
58	0.57	0.56	0.52	0.56	0.53	0.51	0.56	0.55	0.55	109.2	0.0208	3.81	0.062	Y
59	0.51	0.53	0.51	0.53	0.54	0.53	0.56	0.52	0.53	105.7	0.0165	3.12	0.050	N
60	0.53	0.53	0.53	0.51	0.53	0.52	0.57	0.53	0.53	106.3	0.0174	3.28	0.052	Y
61	0.56	0.54	0.49	0.53	0.53	0.51	0.55	0.57	0.53	107.0	0.0273	5.11	0.082	Y
62	0.55	0.54	0.50	0.53	0.54	0.53	0.55	0.58	0.54	107.8	0.0230	4.27	0.069	Y
63	0.56	0.55	0.54	0.55	0.53	0.57	0.56	0.58	0.56	111.2	0.0168	3.02	0.050	Y
64	0.52	0.55	0.50	0.53	0.52	0.50	0.54	0.54	0.53	105.2	0.0180	3.43	0.054	Y

Attachment A Solid SOX MDLs

Attachment A

Northeast Analytical Inc.
Quality Assurance/Quality Control
PCB Congener Specific Method Detection Limit Study

Date: 05/25/2005

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: Soxhlet
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/5/05

65	0.56	0.55	0.52	0.53	0.53	0.56	0.55	0.55	0.54	108.4	0.0133	2.45	0.040	N
66	0.55	0.56	0.51	0.55	0.53	0.55	0.57	0.57	0.55	109.5	0.0190	3.47	0.057	Y
67	0.55	0.56	0.51	0.53	0.51	0.53	0.55	0.57	0.54	107.3	0.0232	4.31	0.069	Y
68	0.54	0.53	0.51	0.50	0.50	0.54	0.53	0.55	0.53	105.3	0.0198	3.76	0.059	Y
69	0.54	0.53	0.48	0.50	0.54	0.49	0.54	0.55	0.52	104.3	0.0260	4.99	0.078	Y
70	0.51	0.52	0.47	0.51	0.57	0.50	0.56	0.51	0.52	103.6	0.0307	5.93	0.092	Y
71	0.54	0.54	0.51	0.53	0.56	0.52	0.59	0.58	0.55	109.3	0.0276	5.05	0.083	Y
72	0.58	0.53	0.52	0.54	0.52	0.50	0.52	0.59	0.54	107.3	0.0308	5.74	0.092	Y
73	0.56	0.54	0.50	0.55	0.54	0.52	0.56	0.59	0.54	108.9	0.028	5.11	0.083	Y
74	0.51	0.52	0.47	0.51	0.57	0.50	0.56	0.51	0.52	103.6	0.0307	5.93	0.092	Y
75	0.55	0.54	0.50	0.53	0.54	0.53	0.55	0.58	0.54	107.8	0.0230	4.27	0.069	Y
76	0.56	0.54	0.49	0.52	0.52	0.49	0.51	0.57	0.53	105.2	0.030	5.78	0.091	Y
77	0.55	0.53	0.52	0.54	0.57	0.53	0.55	0.55	0.54	108.3	0.0151	2.79	0.045	N
78	0.53	0.56	0.52	0.50	0.52	0.50	0.57	0.55	0.53	106.2	0.0285	5.36	0.085	Y
79	0.55	0.54	0.50	0.56	0.56	0.48	0.51	0.54	0.53	105.9	0.0306	5.78	0.092	Y
80	0.55	0.50	0.47	0.56	0.52	0.49	0.52	0.57	0.52	104.3	0.0358	6.86	0.107	Y
81	0.54	0.54	0.51	0.51	0.52	0.53	0.53	0.56	0.53	106.3	0.016	3.10	0.049	N
82	0.56	0.55	0.52	0.53	0.55	0.55	0.55	0.55	0.54	108.8	0.0143	2.63	0.043	N
83	0.54	0.54	0.54	0.53	0.56	0.50	0.56	0.57	0.54	108.5	0.0212	3.92	0.064	Y
84	0.59	0.55	0.51	0.55	0.55	0.49	0.55	0.64	0.55	110.5	0.0467	8.46	0.140	Y
85	0.58	0.57	0.50	0.54	0.57	0.56	0.55	0.64	0.56	112.6	0.0393	6.98	0.118	Y
86	0.57	0.55	0.51	0.54	0.55	0.54	0.55	0.54	0.55	109.2	0.0167	3.05	0.050	N
87	0.56	0.52	0.48	0.53	0.52	0.51	0.54	0.53	0.52	104.7	0.0242	4.62	0.073	Y
88	0.56	0.55	0.54	0.55	0.53	0.57	0.56	0.58	0.56	111.2	0.0168	3.02	0.050	Y
89	0.53	0.55	0.48	0.54	0.53	0.53	0.57	0.57	0.54	107.8	0.0276	5.12	0.083	Y
90	0.56	0.54	0.55	0.53	0.57	0.54	0.55	0.58	0.55	110.8	0.0183	3.30	0.055	Y
91	0.56	0.54	0.49	0.53	0.53	0.51	0.55	0.57	0.53	107.0	0.0273	5.11	0.082	Y
92	0.55	0.50	0.47	0.56	0.52	0.49	0.52	0.57	0.52	104.3	0.0358	6.86	0.107	Y
93	0.57	0.56	0.52	0.56	0.53	0.51	0.56	0.55	0.55	109.2	0.0208	3.81	0.062	Y
94	0.54	0.53	0.51	0.50	0.50	0.54	0.53	0.55	0.53	105.3	0.0198	3.76	0.059	Y
95	0.56	0.55	0.49	0.56	0.55	0.52	0.54	0.56	0.54	108.3	0.0247	4.56	0.074	Y
96	0.51	0.53	0.51	0.53	0.54	0.53	0.56	0.52	0.53	105.7	0.0165	3.12	0.050	N

Attachment A Solid SOX MDLs

Attachment A

Northeast Analytical Inc.
Quality Assurance/Quality Control
PCB Congener Specific Method Detection Limit Study

Date: 05/25/2005

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: Soxhlet
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/5/05

97	0.57	0.55	0.51	0.54	0.55	0.54	0.55	0.54	0.55	109.2	0.0167	3.05	0.050	N
98	0.55	0.56	0.51	0.53	0.51	0.53	0.55	0.57	0.54	107.3	0.0232	4.31	0.069	Y
99	0.58	0.53	0.48	0.55	0.52	0.57	0.57	0.55	0.55	109.1	0.0317	5.81	0.095	Y
100	0.56	0.55	0.49	0.56	0.55	0.52	0.54	0.56	0.54	108.3	0.0247	4.56	0.074	Y
101	0.56	0.54	0.55	0.53	0.57	0.54	0.55	0.58	0.55	110.8	0.0183	3.30	0.055	Y
102	0.57	0.56	0.52	0.56	0.53	0.51	0.56	0.55	0.55	109.2	0.0208	3.81	0.062	Y
103	0.58	0.53	0.52	0.54	0.52	0.50	0.52	0.59	0.54	107.3	0.0308	5.74	0.092	Y
104	0.52	0.54	0.52	0.53	0.55	0.53	0.54	0.53	0.53	106.9	0.0104	1.95	0.031	N
105	0.53	0.50	0.54	0.55	0.56	0.56	0.56	0.57	0.55	109.3	0.0226	4.13	0.068	Y
106	0.57	0.54	0.52	0.55	0.56	0.52	0.52	0.53	0.54	107.5	0.0197	3.67	0.059	Y
107	0.54	0.55	0.56	0.52	0.55	0.54	0.55	0.56	0.55	109.3	0.0123	2.26	0.037	N
108	0.57	0.55	0.51	0.54	0.55	0.54	0.55	0.54	0.55	109.2	0.0167	3.05	0.050	N
109	0.54	0.55	0.56	0.52	0.55	0.54	0.55	0.56	0.55	109.3	0.0123	2.26	0.037	N
110	0.53	0.54	0.52	0.53	0.52	0.56	0.55	0.55	0.54	107.2	0.0135	2.51	0.040	N
111	0.55	0.53	0.52	0.54	0.57	0.53	0.55	0.55	0.54	108.3	0.0151	2.79	0.045	N
112	0.53	0.55	0.52	0.53	0.54	0.52	0.56	0.52	0.53	106.7	0.015	2.72	0.044	N
113	0.56	0.54	0.55	0.53	0.57	0.54	0.55	0.58	0.55	110.8	0.0183	3.30	0.055	Y
114	0.54	0.55	0.50	0.54	0.54	0.52	0.55	0.57	0.54	107.7	0.0217	4.03	0.065	Y
115	0.53	0.54	0.52	0.53	0.52	0.56	0.55	0.55	0.54	107.2	0.0135	2.51	0.040	N
116	0.58	0.57	0.50	0.54	0.57	0.56	0.55	0.64	0.56	112.6	0.0393	6.98	0.118	Y
117	0.58	0.57	0.50	0.54	0.57	0.56	0.55	0.64	0.56	112.6	0.0393	6.98	0.118	Y
118	0.54	0.56	0.50	0.55	0.52	0.57	0.58	0.55	0.55	109.3	0.0256	4.69	0.077	Y
119	0.57	0.55	0.51	0.54	0.55	0.54	0.55	0.54	0.55	109.2	0.0167	3.05	0.050	N
120	0.52	0.55	0.46	0.56	0.54	0.53	0.56	0.56	0.54	107.0	0.034	6.31	0.101	Y
121	0.54	0.55	0.50	0.56	0.53	0.55	0.56	0.56	0.54	108.5	0.0220	4.05	0.066	Y
122	0.56	0.54	0.52	0.52	0.52	0.51	0.51	0.57	0.53	106.6	0.0219	4.12	0.066	Y
123	0.54	0.54	0.53	0.54	0.56	0.55	0.52	0.57	0.54	109.0	0.0152	2.78	0.045	N
124	0.54	0.55	0.56	0.52	0.55	0.54	0.55	0.56	0.55	109.3	0.0123	2.26	0.037	N
125	0.55	0.57	0.52	0.53	0.56	0.51	0.52	0.60	0.55	109.1	0.0292	5.34	0.087	Y
126	0.54	0.53	0.49	0.54	0.52	0.54	0.56	0.55	0.54	107.2	0.0207	3.87	0.062	Y
127	0.59	0.53	0.52	0.52	0.55	0.55	0.58	0.60	0.55	111.0	0.0310	5.59	0.093	Y
128	0.55	0.54	0.54	0.55	0.52	0.51	0.58	0.58	0.55	109.1	0.023	4.15	0.068	Y

Attachment A Solid SOX MDLs

Attachment A

Northeast Analytical Inc.
Quality Assurance/Quality Control
PCB Congener Specific Method Detection Limit Study

Date: 05/25/2005

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: Soxhlet
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/5/05

129	0.56	0.51	0.50	0.51	0.56	0.49	0.58	0.52	0.53	105.7	0.0325	6.16	0.098	Y
130	0.56	0.52	0.54	0.51	0.49	0.47	0.55	0.53	0.52	104.6	0.029	5.60	0.088	Y
131	0.52	0.53	0.53	0.53	0.55	0.53	0.53	0.54	0.53	106.4	0.0103	1.94	0.031	N
132	0.55	0.53	0.50	0.56	0.56	0.58	0.56	0.58	0.55	110.5	0.0253	4.58	0.076	Y
133	0.53	0.55	0.55	0.52	0.50	0.53	0.55	0.55	0.53	106.8	0.0190	3.55	0.057	Y
134	0.55	0.53	0.50	0.53	0.54	0.49	0.57	0.56	0.53	106.8	0.0281	5.27	0.084	Y
135	0.53	0.53	0.49	0.53	0.55	0.55	0.57	0.56	0.54	107.9	0.0249	4.62	0.075	Y
136	0.54	0.54	0.54	0.53	0.56	0.50	0.56	0.57	0.54	108.5	0.0212	3.92	0.064	Y
137	0.54	0.53	0.50	0.53	0.50	0.49	0.54	0.54	0.52	104.5	0.0221	4.22	0.066	Y
138	0.56	0.51	0.50	0.51	0.56	0.49	0.58	0.52	0.53	105.7	0.0325	6.16	0.098	Y
139	0.56	0.55	0.53	0.52	0.55	0.53	0.56	0.57	0.55	109.3	0.0186	3.39	0.056	Y
140	0.56	0.55	0.53	0.52	0.55	0.53	0.56	0.57	0.55	109.3	0.0186	3.39	0.056	Y
141	0.55	0.54	0.50	0.53	0.55	0.49	0.57	0.57	0.54	107.6	0.0296	5.50	0.089	Y
142	0.52	0.53	0.53	0.53	0.55	0.53	0.53	0.54	0.53	106.4	0.0103	1.94	0.031	N
143	0.55	0.53	0.50	0.53	0.54	0.49	0.57	0.56	0.53	106.8	0.0281	5.27	0.084	Y
144	0.54	0.54	0.49	0.53	0.54	0.51	0.56	0.56	0.53	106.8	0.0257	4.80	0.077	Y
145	0.54	0.55	0.52	0.56	0.49	0.57	0.55	0.53	0.54	107.8	0.0265	4.91	0.079	Y
146	0.53	0.50	0.54	0.55	0.56	0.56	0.56	0.57	0.55	109.3	0.0226	4.13	0.068	Y
147	0.54	0.54	0.50	0.54	0.56	0.50	0.57	0.55	0.54	107.7	0.0254	4.71	0.076	Y
148	0.56	0.55	0.52	0.53	0.55	0.55	0.55	0.55	0.54	108.8	0.0143	2.63	0.043	N
149	0.54	0.54	0.50	0.54	0.56	0.50	0.57	0.55	0.54	107.7	0.0254	4.71	0.076	Y
150	0.56	0.54	0.55	0.53	0.57	0.54	0.55	0.58	0.55	110.8	0.0183	3.30	0.055	Y
151	0.55	0.53	0.52	0.54	0.57	0.53	0.55	0.55	0.54	108.3	0.0151	2.79	0.045	N
152	0.53	0.53	0.50	0.54	0.52	0.51	0.54	0.54	0.53	105.2	0.0143	2.73	0.043	N
153	0.56	0.51	0.52	0.54	0.56	0.51	0.56	0.56	0.54	108.0	0.0237	4.40	0.071	Y
154	0.53	0.53	0.49	0.53	0.55	0.55	0.57	0.56	0.54	107.9	0.0249	4.62	0.075	Y
155	0.53	0.53	0.50	0.54	0.52	0.51	0.54	0.54	0.53	105.2	0.0143	2.73	0.043	N
156	0.58	0.55	0.51	0.55	0.57	0.54	0.55	0.59	0.56	111.1	0.0231	4.15	0.069	Y
157	0.58	0.55	0.51	0.55	0.57	0.54	0.55	0.59	0.56	111.1	0.0231	4.15	0.069	Y
158	0.56	0.55	0.51	0.52	0.55	0.55	0.57	0.60	0.55	110.0	0.0288	5.23	0.086	Y
159	0.53	0.54	0.53	0.51	0.55	0.54	0.53	0.54	0.53	106.6	0.0122	2.29	0.037	N
160	0.55	0.55	0.48	0.51	0.52	0.49	0.57	0.53	0.52	104.8	0.0305	5.82	0.091	Y

Attachment A Solid SOX MDLs

Attachment A

Northeast Analytical Inc.
Quality Assurance/Quality Control
PCB Congener Specific Method Detection Limit Study

Date: 05/25/2005

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: Soxhlet
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/5/05

161	0.58	0.53	0.54	0.52	0.51	0.54	0.55	0.56	0.54	108.2	0.0234	4.32	0.070	Y
162	0.59	0.56	0.54	0.55	0.55	0.55	0.55	0.59	0.56	111.7	0.0182	3.25	0.054	Y
163	0.56	0.51	0.50	0.51	0.56	0.49	0.58	0.52	0.53	105.7	0.0325	6.16	0.098	Y
164	0.54	0.53	0.50	0.54	0.54	0.52	0.56	0.59	0.54	108.0	0.0271	5.01	0.081	Y
165	0.56	0.52	0.54	0.52	0.52	0.56	0.56	0.57	0.54	109.0	0.0210	3.86	0.063	Y
166	0.55	0.54	0.54	0.55	0.52	0.51	0.58	0.58	0.55	109.1	0.023	4.15	0.068	Y
167	0.58	0.51	0.48	0.56	0.50	0.47	0.55	0.57	0.53	105.4	0.0447	8.47	0.134	Y
168	0.56	0.51	0.52	0.54	0.56	0.51	0.56	0.56	0.54	108.0	0.0237	4.40	0.071	Y
169	0.54	0.54	0.48	0.56	0.54	0.55	0.54	0.54	0.53	106.8	0.0232	4.35	0.070	Y
170	0.57	0.53	0.48	0.54	0.51	0.54	0.53	0.56	0.53	106.3	0.0286	5.38	0.086	Y
171	0.54	0.53	0.56	0.57	0.56	0.56	0.54	0.57	0.55	110.9	0.0155	2.79	0.046	N
172	0.54	0.54	0.48	0.55	0.53	0.51	0.55	0.53	0.53	105.9	0.0231	4.37	0.069	Y
173	0.54	0.53	0.56	0.57	0.56	0.56	0.54	0.57	0.55	110.9	0.0155	2.79	0.046	N
174	0.54	0.53	0.49	0.52	0.53	0.54	0.52	0.56	0.53	105.8	0.0206	3.90	0.062	Y
175	0.51	0.52	0.50	0.54	0.50	0.50	0.54	0.49	0.51	102.5	0.0194	3.79	0.058	Y
176	0.57	0.56	0.48	0.53	0.52	0.53	0.54	0.56	0.54	107.4	0.0302	5.63	0.091	Y
177	0.53	0.52	0.50	0.53	0.56	0.47	0.55	0.54	0.53	105.1	0.0275	5.23	0.082	Y
178	0.55	0.53	0.51	0.56	0.48	0.53	0.56	0.54	0.53	106.6	0.026	4.80	0.077	Y
179	0.54	0.54	0.54	0.52	0.54	0.48	0.56	0.54	0.53	106.6	0.025	4.74	0.076	Y
180	0.58	0.56	0.54	0.51	0.53	0.56	0.57	0.56	0.55	110.4	0.0225	4.08	0.068	Y
181	0.58	0.54	0.54	0.55	0.51	0.49	0.56	0.57	0.54	108.5	0.0302	5.56	0.091	Y
182	0.54	0.53	0.49	0.54	0.52	0.54	0.56	0.55	0.54	107.2	0.0207	3.87	0.062	Y
183	0.55	0.54	0.52	0.53	0.51	0.54	0.52	0.51	0.53	105.7	0.0140	2.64	0.042	N
184	0.56	0.52	0.54	0.52	0.52	0.56	0.56	0.57	0.54	109.0	0.0210	3.86	0.063	Y
185	0.52	0.54	0.51	0.53	0.55	0.50	0.52	0.56	0.53	105.5	0.0197	3.74	0.059	Y
186	0.56	0.56	0.54	0.55	0.52	0.56	0.52	0.56	0.55	109.2	0.0164	3.00	0.049	N
187	0.58	0.53	0.54	0.52	0.54	0.51	0.58	0.57	0.55	109.5	0.0257	4.70	0.077	Y
188	0.54	0.51	0.53	0.53	0.53	0.54	0.54	0.57	0.54	107.4	0.0178	3.32	0.053	Y
189	0.57	0.51	0.54	0.52	0.55	0.49	0.53	0.57	0.54	107.0	0.0269	5.03	0.081	Y
190	0.56	0.54	0.49	0.54	0.54	0.50	0.53	0.54	0.53	105.9	0.0232	4.38	0.070	Y
191	0.56	0.54	0.47	0.53	0.52	0.52	0.54	0.55	0.53	105.9	0.0272	5.13	0.081	Y
192	0.55	0.54	0.52	0.53	0.53	0.50	0.52	0.55	0.53	105.8	0.0182	3.44	0.055	Y

Attachment A Solid SOX MDLs

Attachment A

Northeast Analytical Inc.
Quality Assurance/Quality Control
PCB Congener Specific Method Detection Limit Study

Date: 05/25/2005

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: Solid
Extraction: Soxhlet
Spike conc: 0.50 ng/g per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/5/05

193	0.58	0.56	0.54	0.51	0.53	0.56	0.57	0.56	0.55	110.4	0.0225	4.08	0.068	Y
194	0.54	0.51	0.49	0.52	0.54	0.53	0.53	0.53	0.52	104.6	0.0174	3.33	0.052	Y
195	0.54	0.56	0.49	0.52	0.52	0.53	0.53	0.54	0.53	105.6	0.0188	3.57	0.056	Y
196	0.57	0.55	0.50	0.53	0.56	0.53	0.52	0.55	0.54	107.7	0.0222	4.12	0.067	Y
197	0.58	0.55	0.51	0.55	0.57	0.54	0.55	0.59	0.56	111.1	0.0231	4.15	0.069	Y
198	0.56	0.54	0.54	0.54	0.54	0.55	0.55	0.59	0.55	110.2	0.0166	3.02	0.050	N
199	0.56	0.54	0.54	0.54	0.54	0.55	0.55	0.59	0.55	110.2	0.0166	3.02	0.050	N
200	0.56	0.54	0.50	0.53	0.56	0.52	0.56	0.55	0.54	108.1	0.0238	4.41	0.071	Y
201	0.55	0.53	0.50	0.51	0.57	0.54	0.51	0.56	0.53	106.8	0.0271	5.07	0.081	Y
202	0.59	0.56	0.54	0.55	0.55	0.55	0.55	0.59	0.56	111.7	0.0182	3.25	0.054	Y
203	0.57	0.55	0.52	0.52	0.51	0.49	0.56	0.55	0.53	106.8	0.025	4.75	0.076	Y
204	0.55	0.53	0.46	0.53	0.52	0.49	0.54	0.61	0.53	105.5	0.0427	8.09	0.128	Y
205	0.55	0.53	0.52	0.53	0.52	0.52	0.54	0.56	0.53	106.7	0.0160	3.00	0.048	N
206	0.56	0.55	0.50	0.55	0.49	0.52	0.52	0.54	0.53	105.8	0.024	4.46	0.071	Y
207	0.57	0.51	0.54	0.52	0.55	0.49	0.53	0.57	0.54	107.0	0.0269	5.03	0.081	Y
208	0.53	0.55	0.51	0.55	0.56	0.54	0.55	0.53	0.54	108.1	0.0159	2.94	0.048	N
209	0.53	0.53	0.52	0.52	0.52	0.53	0.54	0.55	0.53	105.8	0.0106	2.01	0.032	N

Attachment A Water CLLE MDLs

ATTACHMENT A

Northeast Analytical Inc.

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Date: 06/21/2005

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: 1L Water
Extraction: CLLE
Spike conc: 1.00 ng/L per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/22/05

NEA Sample ID	050421W1	050421W2	050421W3	050421W4	050421W5	050421W6	050421W7	AVERAGE CONCENTRATION	AVERAGE PERCENT RECOVERY	STANDARD DEVIATION (ng/L)	% RELATIVE STANDARD DEVIATION	MDL (2) (ng/L)	Valid[2]
Congener IUPAC#	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	(%)		(%)		
1 (1)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0	0.000	#DIV/0!	0.000	
2 (1)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0	0.0000	#DIV/0!	0.000	
3 (1)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0	0.0000	#DIV/0!	0.000	
4	1.11	0.98	1.08	1.13	1.01	0.99	0.98	1.03	103.4	0.064	6.20	0.192	Y
5	0.93	0.96	0.98	1.02	0.98	0.96	1.02	0.98	97.7	0.0320	3.27	0.096	N
6	1.07	0.97	1.01	1.08	1.02	1.10	1.10	1.05	104.9	0.0482	4.60	0.145	Y
7	0.92	1.09	0.99	1.01	0.96	1.08	1.03	1.02	101.6	0.0582	5.73	0.174	Y
8	1.05	1.10	1.05	1.05	1.03	1.03	0.97	1.03	103.3	0.0409	3.96	0.122	Y
9	0.97	0.94	1.10	1.01	1.04	1.01	1.05	1.02	102.3	0.0547	5.35	0.164	Y
10	1.04	0.89	0.98	1.04	1.05	1.00	0.96	0.99	98.9	0.0555	5.61	0.166	Y
11	0.99	0.95	0.98	1.02	0.90	0.95	1.06	0.99	98.9	0.0539	5.45	0.162	Y
12	1.04	0.99	1.00	1.01	1.02	0.99	1.06	1.00	100.4	0.042	4.18	0.126	Y
13	1.04	0.99	1.00	1.01	1.02	0.99	1.06	1.00	100.4	0.042	4.18	0.126	Y
14	1.05	0.96	1.07	1.06	1.07	1.01	1.03	1.02	102.3	0.049	4.83	0.148	Y
15	1.01	0.97	0.98	1.05	1.02	0.97	0.99	0.99	99.1	0.0348	3.51	0.104	Y
16	1.03	0.98	1.00	0.95	1.05	1.08	0.97	1.01	100.9	0.042	4.15	0.126	Y
17	0.99	0.95	0.98	1.02	0.90	0.95	1.06	0.99	98.9	0.0539	5.45	0.162	Y
18	1.03	1.04	1.06	0.99	1.05	0.99	1.05	1.03	102.8	0.029	2.81	0.087	N
19	0.96	1.06	0.98	1.04	1.05	1.09	0.99	1.03	102.8	0.0460	4.48	0.138	Y
20	1.01	1.01	1.01	1.07	1.09	1.12	1.08	1.05	105.4	0.0438	4.16	0.131	Y
21	0.96	1.01	0.92	1.02	0.96	1.04	1.03	0.99	99.4	0.0406	4.08	0.122	Y
22	1.01	0.98	0.99	0.98	1.05	1.04	0.99	1.00	100.5	0.029	2.91	0.088	N
23	0.91	1.03	1.01	1.05	0.94	0.99	1.00	0.98	98.2	0.0488	4.97	0.146	Y
24	1.04	0.99	1.00	1.01	1.02	0.99	1.06	1.00	100.4	0.042	4.18	0.126	Y
25	0.97	1.03	1.00	0.99	1.05	1.09	1.05	1.02	102.5	0.038	3.69	0.113	Y
26	0.95	0.96	1.04	1.00	0.96	1.02	1.08	1.00	99.8	0.044	4.42	0.132	Y
27	0.97	1.01	1.03	1.10	1.11	1.07	1.02	1.04	103.7	0.0521	5.02	0.156	Y
28	1.01	1.01	1.01	1.07	1.09	1.12	1.08	1.05	105.4	0.0438	4.16	0.131	Y
29	0.95	0.96	1.04	1.00	0.96	1.02	1.08	1.00	99.8	0.044	4.42	0.132	Y
30	1.06	1.07	1.05	1.05	1.06	1.01	1.08	1.03	103.4	0.0597	5.77	0.179	Y
31	1.03	0.99	1.02	1.01	0.96	1.08	0.94	1.00	100.1	0.0442	4.41	0.132	Y
32	0.95	1.04	1.01	1.02	0.97	1.02	0.95	1.00	99.8	0.037	3.66	0.110	Y

Attachment A Water CLLE MDLs

ATTACHMENT A

Northeast Analytical Inc.

Date: 06/21/2005

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: 1L Water
Extraction: CLLE
Spike conc: 1.00 ng/L per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/22/05

33	0.99	1.01	1.03	1.08	1.01	1.09	0.92	1.02	101.8	0.053	5.25	0.160	Y
34	0.97	0.96	1.04	0.97	1.07	1.05	1.05	1.02	101.6	0.0428	4.21	0.128	Y
35	0.95	0.90	1.00	1.04	1.00	1.11	1.04	1.00	99.8	0.0663	6.64	0.199	Y
36	1.08	0.93	1.06	1.13	1.11	1.00	1.03	1.04	104.1	0.0657	6.31	0.197	Y
37	0.96	1.04	1.06	0.98	0.95	0.96	1.05	1.01	100.7	0.0483	4.80	0.145	Y
38	1.03	0.99	0.97	1.05	1.02	1.04	1.05	1.02	101.6	0.0312	3.07	0.094	N
39	1.00	0.94	0.97	1.02	1.04	1.03	1.09	1.01	100.7	0.0498	4.95	0.149	Y
40	0.95	0.90	1.00	1.04	1.00	1.11	1.04	1.00	99.8	0.0663	6.64	0.199	Y
41	0.97	0.92	1.05	1.07	1.01	1.09	0.92	1.00	100.0	0.0663	6.63	0.199	Y
42	1.03	0.99	0.97	1.05	1.02	1.04	1.05	1.02	101.6	0.0312	3.07	0.094	N
43	0.99	0.99	0.93	1.07	0.96	1.11	0.95	1.01	100.9	0.0655	6.49	0.196	Y
44	1.00	0.94	0.97	1.02	1.04	1.03	1.09	1.01	100.7	0.0498	4.95	0.149	Y
45	1.01	1.01	1.01	1.07	1.09	1.12	1.08	1.05	105.4	0.0438	4.16	0.131	Y
46	1.12	1.01	0.99	1.05	0.98	1.08	1.09	1.04	104.2	0.0516	4.96	0.155	Y
47	1.00	0.94	0.97	1.02	1.04	1.03	1.09	1.01	100.7	0.0498	4.95	0.149	Y
48	0.97	0.98	1.01	1.08	0.99	1.03	0.94	1.00	100.4	0.0414	4.12	0.124	Y
49	1.08	0.93	1.06	1.13	1.11	1.00	1.03	1.04	104.1	0.0657	6.31	0.197	Y
50	0.95	0.94	0.99	1.00	1.08	1.03	1.05	1.02	101.5	0.0536	5.28	0.161	Y
51	1.01	1.01	1.01	1.07	1.09	1.12	1.08	1.05	105.4	0.0438	4.16	0.131	Y
52	1.07	1.02	0.98	0.97	1.03	1.06	1.08	1.03	102.6	0.040	3.92	0.121	Y
53	0.97	1.03	1.00	0.99	1.05	1.09	1.05	1.02	102.5	0.038	3.69	0.113	Y
54	1.02	0.96	0.98	1.03	0.95	1.01	0.97	1.01	100.9	0.0640	6.34	0.192	Y
55	0.96	1.03	1.07	1.09	0.98	0.99	1.04	1.02	101.7	0.0479	4.71	0.143	Y
56	0.94	1.00	1.00	1.13	0.93	1.02	1.02	1.02	101.5	0.0651	6.42	0.195	Y
57	1.08	0.94	0.99	1.06	1.07	1.05	1.05	1.03	103.2	0.0480	4.65	0.144	Y
58	0.99	0.97	0.99	1.06	1.07	1.03	0.98	1.02	101.6	0.0393	3.87	0.118	Y
59	1.06	0.98	0.99	1.03	1.04	1.07	0.89	1.01	101.5	0.0615	6.06	0.184	Y
60	1.07	0.94	1.04	1.07	0.97	1.03	0.91	1.01	101.0	0.0611	6.05	0.183	Y
61	0.97	1.00	1.01	1.07	0.96	1.05	0.96	1.00	99.8	0.0458	4.59	0.137	Y
62	0.96	0.99	0.99	0.99	0.93	0.96	0.94	0.97	97.1	0.0279	2.87	0.084	N
63	0.98	1.01	1.06	1.14	1.08	0.97	0.98	1.02	102.4	0.0625	6.11	0.187	Y
64	0.92	1.00	1.00	1.08	0.96	1.02	1.03	1.01	100.8	0.0499	4.95	0.150	Y

Attachment A Water CLLE MDLs

ATTACHMENT A

Northeast Analytical Inc.

Date: 06/21/2005

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: 1L Water
Extraction: CLLE
Spike conc: 1.00 ng/L per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/22/05

65	1.00	0.94	0.97	1.02	1.04	1.03	1.09	1.01	100.7	0.0498	4.95	0.149	Y
66	1.00	1.01	1.04	0.96	1.06	1.01	0.93	1.00	99.9	0.0418	4.19	0.125	Y
67	1.03	1.00	0.99	1.09	1.04	1.05	0.99	1.03	102.9	0.0344	3.34	0.103	Y
68	0.98	0.97	0.98	0.99	0.97	0.97	0.99	0.98	97.7	0.0117	1.20	0.035	N
69	1.04	0.97	1.01	1.06	1.06	0.97	0.97	1.01	101.4	0.0391	3.85	0.117	Y
70	0.96	1.09	1.00	1.00	1.02	1.01	0.96	1.01	100.6	0.0406	4.03	0.122	Y
71	0.95	0.90	1.00	1.04	1.00	1.11	1.04	1.00	99.8	0.0663	6.64	0.199	Y
72	0.97	1.01	1.00	1.00	0.97	1.04	1.08	1.01	101.3	0.0370	3.66	0.111	Y
73	1.03	0.98	1.06	1.03	1.07	1.06	1.08	1.04	104.1	0.033	3.20	0.100	N
74	0.96	1.09	1.00	1.00	1.02	1.01	0.96	1.01	100.6	0.0406	4.03	0.122	Y
75	0.96	0.99	0.99	0.99	0.93	0.96	0.94	0.97	97.1	0.0279	2.87	0.084	N
76	0.98	1.08	1.03	1.09	1.04	0.97	1.03	1.03	103.4	0.041	3.93	0.122	Y
77	1.06	0.99	1.04	1.03	0.95	1.06	1.04	1.02	102.4	0.0370	3.62	0.111	Y
78	0.98	0.97	1.01	1.03	0.95	0.99	1.00	0.99	98.8	0.0275	2.78	0.082	N
79	0.94	0.87	1.00	1.00	0.95	1.00	0.90	0.96	95.8	0.0513	5.35	0.154	Y
80	0.99	1.03	1.01	1.11	0.93	0.97	1.01	1.01	100.6	0.0511	5.08	0.153	Y
81	0.99	1.10	1.02	1.00	1.02	0.97	0.92	1.01	100.8	0.052	5.19	0.157	Y
82	0.96	1.03	1.03	1.08	1.12	1.01	0.92	1.02	102.3	0.0613	5.99	0.184	Y
83	1.00	1.05	1.01	1.01	1.02	0.99	1.01	1.02	101.6	0.0227	2.24	0.068	N
84	0.96	1.01	1.02	1.07	0.99	1.06	1.02	1.02	101.9	0.0345	3.39	0.103	Y
85	1.07	1.09	1.11	1.14	1.04	0.98	1.08	1.06	106.3	0.0535	5.03	0.160	Y
86	1.05	1.05	1.12	1.06	1.04	1.04	1.08	1.06	106.3	0.0281	2.64	0.084	N
87	1.00	1.01	0.94	1.02	0.92	1.03	1.08	0.99	98.8	0.0616	6.23	0.185	Y
88	0.98	1.01	1.06	1.14	1.08	0.97	0.98	1.02	102.4	0.0625	6.11	0.187	Y
89	0.96	1.03	1.07	1.09	0.98	0.99	1.04	1.02	101.7	0.0479	4.71	0.143	Y
90	1.06	1.09	1.14	1.10	1.08	1.10	1.10	1.10	109.6	0.0226	2.06	0.068	N
91	0.97	1.00	1.01	1.07	0.96	1.05	0.96	1.00	99.8	0.0458	4.59	0.137	Y
92	0.99	1.03	1.01	1.11	0.93	0.97	1.01	1.01	100.6	0.0511	5.08	0.153	Y
93	0.99	0.97	0.99	1.06	1.07	1.03	0.98	1.02	101.6	0.0393	3.87	0.118	Y
94	0.98	0.97	0.98	0.99	0.97	0.97	0.99	0.98	97.7	0.0117	1.20	0.035	N
95	1.04	1.05	1.01	1.04	1.05	1.09	1.10	1.05	105.2	0.0300	2.85	0.090	N
96	1.06	0.98	0.99	1.03	1.04	1.07	0.89	1.01	101.5	0.0615	6.06	0.184	Y

Attachment A Water CLLE MDLs

ATTACHMENT A

Northeast Analytical Inc.

Date: 06/21/2005

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: 1L Water
Extraction: CLLE
Spike conc: 1.00 ng/L per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/22/05

97	1.05	1.05	1.12	1.06	1.04	1.04	1.08	1.06	106.3	0.0281	2.64	0.084	N
98	1.03	1.00	0.99	1.09	1.04	1.05	0.99	1.03	102.9	0.0344	3.34	0.103	Y
99	1.00	0.95	1.00	1.10	1.08	1.04	0.99	1.02	102.1	0.0505	4.94	0.151	Y
100	1.04	1.05	1.01	1.04	1.05	1.09	1.10	1.05	105.2	0.0300	2.85	0.090	N
101	1.06	1.09	1.14	1.10	1.08	1.10	1.10	1.10	109.6	0.0226	2.06	0.068	N
102	0.99	0.97	0.99	1.06	1.07	1.03	0.98	1.02	101.6	0.0393	3.87	0.118	Y
103	0.97	1.01	1.00	1.00	0.97	1.04	1.08	1.01	101.3	0.0370	3.66	0.111	Y
104	1.06	1.01	0.96	1.09	1.04	1.07	0.98	1.03	102.7	0.0463	4.51	0.139	Y
105	0.91	0.90	1.00	1.00	1.00	1.00	1.07	0.99	98.9	0.0568	5.74	0.170	Y
106	1.00	0.97	1.02	0.95	1.04	0.95	1.04	0.98	98.5	0.0492	5.00	0.148	Y
107	0.94	0.97	1.05	0.99	1.00	1.04	0.93	0.99	98.9	0.0414	4.19	0.124	Y
108	0.94	0.97	1.05	0.99	1.00	1.04	0.93	0.99	98.9	0.0414	4.19	0.124	Y
109	1.05	1.05	1.12	1.06	1.04	1.04	1.08	1.06	106.3	0.0281	2.64	0.084	N
110	0.98	1.06	1.01	0.99	1.01	0.97	0.99	1.01	100.6	0.0303	3.01	0.091	N
111	1.06	0.99	1.04	1.03	0.95	1.06	1.04	1.02	102.4	0.0370	3.62	0.111	Y
112	0.98	0.99	1.00	1.04	0.90	1.06	1.01	1.00	100.2	0.049	4.94	0.148	Y
113	1.06	1.09	1.14	1.10	1.08	1.10	1.10	1.10	109.6	0.0226	2.06	0.068	N
114	1.07	1.01	1.03	1.03	1.06	0.98	1.03	1.02	102.2	0.0387	3.79	0.116	Y
115	0.98	1.06	1.01	0.99	1.01	0.97	0.99	1.01	100.6	0.0303	3.01	0.091	N
116	1.07	1.09	1.11	1.14	1.04	0.98	1.08	1.06	106.3	0.0535	5.03	0.160	Y
117	1.07	1.09	1.11	1.14	1.04	0.98	1.08	1.06	106.3	0.0535	5.03	0.160	Y
118	0.97	0.92	1.09	1.07	1.04	1.08	1.09	1.04	104.1	0.0620	5.96	0.186	Y
119	1.05	1.05	1.12	1.06	1.04	1.04	1.08	1.06	106.3	0.0281	2.64	0.084	N
120	1.07	1.00	0.99	0.98	0.98	1.09	0.97	1.00	100.3	0.050	4.95	0.149	Y
121	1.00	1.02	1.00	1.12	1.01	1.14	1.02	1.04	103.6	0.0592	5.71	0.178	Y
122	1.02	0.94	1.08	0.99	1.06	1.03	1.09	1.02	102.5	0.0501	4.89	0.150	Y
123	0.97	1.04	1.03	0.97	1.00	1.01	0.83	0.98	98.2	0.0666	6.78	0.200	Y
124	0.94	0.97	1.05	0.99	1.00	1.04	0.93	0.99	98.9	0.0414	4.19	0.124	Y
125	0.89	1.02	1.04	1.02	0.97	1.04	1.06	1.01	100.7	0.0538	5.34	0.161	Y
126	0.98	0.94	1.00	1.02	1.00	1.00	0.96	0.99	98.9	0.0274	2.77	0.082	N
127	0.99	1.02	1.00	1.06	1.01	0.97	1.00	1.01	101.4	0.0374	3.69	0.112	Y
128	0.97	0.98	1.03	1.03	0.97	1.00	0.96	0.99	99.2	0.026	2.61	0.078	N

Attachment A Water CLLE MDLs

ATTACHMENT A

Northeast Analytical Inc.

Date: 06/21/2005

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: 1L Water
Extraction: CLLE
Spike conc: 1.00 ng/L per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/22/05

129	1.02	1.04	1.00	1.01	1.02	0.96	1.03	1.01	101.0	0.0249	2.47	0.075	N
130	1.08	1.01	1.04	0.97	0.93	1.01	1.03	1.01	100.5	0.047	4.68	0.141	Y
131	0.95	0.92	1.00	0.95	1.00	1.04	0.90	0.96	96.0	0.0478	4.98	0.143	Y
132	0.98	1.08	1.02	1.08	0.98	1.07	1.08	1.04	103.7	0.0432	4.17	0.130	Y
133	0.97	0.94	1.05	0.97	0.99	1.00	1.06	0.99	99.4	0.0415	4.18	0.125	Y
134	0.95	1.10	1.07	1.08	1.00	0.98	0.98	1.04	103.5	0.0645	6.23	0.193	Y
135	1.04	0.98	1.03	1.04	1.06	1.04	1.07	1.03	102.5	0.0413	4.03	0.124	Y
136	1.00	1.05	1.01	1.01	1.02	0.99	1.01	1.02	101.6	0.0227	2.24	0.068	N
137	1.02	0.96	1.07	0.98	0.95	1.02	1.03	0.99	99.2	0.0524	5.28	0.157	Y
138	1.02	1.04	1.00	1.01	1.02	0.96	1.03	1.01	101.0	0.0249	2.47	0.075	N
139	0.98	1.05	1.11	1.08	0.97	1.02	1.03	1.02	102.4	0.0525	5.13	0.157	Y
140	0.98	1.05	1.11	1.08	0.97	1.02	1.03	1.02	102.4	0.0525	5.13	0.157	Y
141	0.87	0.90	1.06	1.03	0.97	0.97	1.00	0.97	97.3	0.0619	6.36	0.185	Y
142	0.95	0.92	1.00	0.95	1.00	1.04	0.90	0.96	96.0	0.0478	4.98	0.143	Y
143	0.95	1.10	1.07	1.08	1.00	0.98	0.98	1.04	103.5	0.0645	6.23	0.193	Y
144	1.08	0.97	1.03	1.01	1.04	1.04	0.91	1.01	100.8	0.0534	5.30	0.160	Y
145	1.01	1.00	1.00	1.09	1.01	0.96	1.10	1.04	103.8	0.0616	5.93	0.185	Y
146	0.91	0.90	1.00	1.00	1.00	1.00	1.07	0.99	98.9	0.0568	5.74	0.170	Y
147	1.08	1.01	1.15	1.11	1.03	1.01	0.98	1.06	105.9	0.0597	5.63	0.179	Y
148	0.96	1.03	1.03	1.08	1.12	1.01	0.92	1.02	102.3	0.0613	5.99	0.184	Y
149	1.08	1.01	1.15	1.11	1.03	1.01	0.98	1.06	105.9	0.0597	5.63	0.179	Y
150	1.06	1.09	1.14	1.10	1.08	1.10	1.10	1.10	109.6	0.0226	2.06	0.068	N
151	1.06	0.99	1.04	1.03	0.95	1.06	1.04	1.02	102.4	0.0370	3.62	0.111	Y
152	0.97	1.07	0.98	1.06	1.03	1.04	0.91	1.01	101.2	0.0556	5.49	0.167	Y
153	0.96	0.99	1.04	1.02	1.00	1.12	1.07	1.03	102.6	0.0485	4.72	0.145	Y
154	1.04	0.98	1.03	1.04	1.06	1.04	1.07	1.03	102.5	0.0413	4.03	0.124	Y
155	0.97	1.07	0.98	1.06	1.03	1.04	0.91	1.01	101.2	0.0556	5.49	0.167	Y
156	0.92	0.99	1.10	0.98	0.93	1.06	1.03	1.00	100.0	0.0621	6.21	0.186	Y
157	0.92	0.99	1.10	0.98	0.93	1.06	1.03	1.00	100.0	0.0621	6.21	0.186	Y
158	0.97	1.06	1.08	1.10	1.01	0.95	1.04	1.04	103.7	0.0555	5.35	0.166	Y
159	0.96	1.02	1.02	1.02	0.98	0.99	1.07	1.01	100.8	0.0347	3.44	0.104	Y
160	0.92	1.05	1.04	1.00	1.01	0.98	0.87	0.98	98.1	0.0615	6.27	0.184	Y

Attachment A Water CLLE MDLs

ATTACHMENT A

Northeast Analytical Inc.

Date: 06/21/2005

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: 1L Water
Extraction: CLLE
Spike conc: 1.00 ng/L per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/22/05

161	0.94	0.95	1.02	0.97	0.91	0.98	1.03	0.98	97.9	0.0425	4.34	0.127	Y
162	1.09	1.00	1.02	0.97	0.93	1.03	1.03	1.02	101.8	0.0501	4.92	0.150	Y
163	1.02	1.04	1.00	1.01	1.02	0.96	1.03	1.01	101.0	0.0249	2.47	0.075	N
164	1.08	1.03	1.06	1.08	1.00	1.03	0.98	1.03	102.7	0.0471	4.59	0.141	Y
165	1.05	0.95	1.05	0.95	1.06	1.11	0.94	1.02	101.9	0.0652	6.39	0.195	Y
166	0.97	0.98	1.03	1.03	0.97	1.00	0.96	0.99	99.2	0.026	2.61	0.078	N
167	0.99	0.96	1.05	1.14	0.96	1.06	0.99	1.02	101.8	0.0619	6.08	0.186	Y
168	0.96	0.99	1.04	1.02	1.00	1.12	1.07	1.03	102.6	0.0485	4.72	0.145	Y
169	0.95	0.91	1.02	0.96	1.00	0.98	0.89	0.95	95.4	0.0453	4.75	0.136	Y
170	1.02	0.88	1.04	0.98	1.00	1.00	1.01	0.99	99.5	0.0509	5.11	0.153	Y
171	0.94	1.02	1.04	1.02	1.02	1.08	0.97	1.02	102.5	0.0535	5.22	0.160	Y
172	1.02	1.00	1.03	1.07	0.93	0.99	0.89	0.99	99.0	0.0573	5.79	0.172	Y
173	0.94	1.02	1.04	1.02	1.02	1.08	0.97	1.02	102.5	0.0535	5.22	0.160	Y
174	1.06	0.93	0.97	1.10	1.08	1.10	1.07	1.04	104.0	0.0645	6.20	0.193	Y
175	0.92	1.06	1.03	1.04	0.93	1.00	0.92	0.98	98.2	0.0579	5.90	0.174	Y
176	1.02	1.01	1.04	1.03	1.04	1.03	0.97	1.01	101.2	0.0293	2.89	0.088	N
177	0.90	0.94	1.00	1.00	1.02	1.02	0.84	0.96	96.3	0.0631	6.55	0.189	Y
178	0.99	1.02	1.08	1.04	0.98	1.02	0.92	1.00	100.4	0.048	4.82	0.145	Y
179	1.01	0.99	1.01	1.10	1.03	0.96	1.06	1.02	102.3	0.042	4.14	0.127	Y
180	1.05	1.02	1.10	1.12	1.08	1.02	1.05	1.06	106.1	0.0390	3.68	0.117	Y
181	0.99	1.07	0.98	1.06	0.96	1.08	1.04	1.03	103.3	0.0489	4.74	0.147	Y
182	0.98	0.94	1.00	1.02	1.00	1.00	0.96	0.99	98.9	0.0274	2.77	0.082	N
183	1.08	0.99	1.03	1.10	0.99	1.00	1.02	1.03	103.4	0.0446	4.31	0.134	Y
184	1.05	0.95	1.05	0.95	1.06	1.11	0.94	1.02	101.9	0.0652	6.39	0.195	Y
185	1.01	0.96	0.96	1.08	1.03	0.95	0.88	0.97	97.5	0.0643	6.60	0.193	Y
186	0.96	0.98	1.05	1.11	1.06	1.01	1.07	1.05	105.0	0.0640	6.10	0.192	Y
187	1.07	1.06	1.04	1.11	1.03	1.10	0.90	1.04	104.0	0.0657	6.32	0.197	Y
188	1.01	1.05	1.09	0.99	1.09	1.01	1.09	1.04	103.9	0.0483	4.65	0.145	Y
189	0.96	1.05	1.03	0.99	1.07	1.04	0.90	1.01	100.7	0.0551	5.47	0.165	Y
190	0.99	0.99	1.00	1.11	1.02	1.00	0.92	1.00	100.2	0.0534	5.33	0.160	Y
191	0.98	1.07	1.04	1.13	1.13	1.03	0.99	1.04	104.5	0.0627	6.00	0.188	Y
192	0.99	0.97	1.03	0.98	0.95	1.00	0.98	1.00	99.5	0.0387	3.89	0.116	Y

Attachment A Water CLLE MDLs

ATTACHMENT A

Northeast Analytical Inc.

Date: 06/21/2005

Quality Assurance/Quality Control

PCB Congener Specific Method Detection Limit Study

Method: Comprehensive Quantitative Congener Specific PCB SW-846 Method 8082
Compound: PCB Congeners
Matrix: 1L Water
Extraction: CLLE
Spike conc: 1.00 ng/L per Congener

Analysis: CQCS PCB : Internal Standard Calibration
Instrument: GC-15 (Agilent Model 6890)
Column: Chrompack : CP-SIL 5/C18-CB, Fused Silica 50m x 0.25mm
Detector: Electron Capture Detector
Date Extracted: 4/22/05

193	1.05	1.02	1.10	1.12	1.08	1.02	1.05	1.06	106.1	0.0390	3.68	0.117	Y
194	0.96	0.93	0.98	1.00	1.04	1.03	0.89	0.98	97.9	0.0530	5.41	0.159	Y
195	0.93	1.05	0.97	0.98	0.94	0.99	1.02	0.98	97.8	0.0426	4.36	0.128	Y
196	0.94	0.99	0.98	0.99	1.01	1.13	1.07	1.02	102.5	0.0629	6.14	0.189	Y
197	0.92	0.99	1.10	0.98	0.93	1.06	1.03	1.00	100.0	0.0621	6.21	0.186	Y
198	1.05	1.06	1.06	1.09	1.02	0.99	0.94	1.03	103.3	0.0472	4.57	0.142	Y
199	1.05	1.06	1.06	1.09	1.02	0.99	0.94	1.03	103.3	0.0472	4.57	0.142	Y
200	0.98	1.04	1.09	1.05	0.99	1.05	0.91	1.01	101.5	0.0551	5.43	0.165	Y
201	0.96	1.03	1.03	1.07	1.08	1.03	1.02	1.03	102.5	0.0413	4.03	0.124	Y
202	1.09	1.00	1.02	0.97	0.93	1.03	1.03	1.02	101.8	0.0501	4.92	0.150	Y
203	1.09	1.08	0.98	0.99	0.99	0.98	1.06	1.01	100.9	0.062	6.10	0.185	Y
204	0.99	1.09	1.04	1.03	0.98	1.08	1.06	1.04	104.0	0.0380	3.65	0.114	Y
205	0.93	1.04	1.06	1.03	1.02	0.99	1.07	1.01	100.9	0.0568	5.63	0.170	Y
206	0.97	0.93	1.09	1.08	0.97	1.01	1.05	1.00	100.4	0.064	6.37	0.192	Y
207	0.96	1.05	1.03	0.99	1.07	1.04	0.90	1.01	100.7	0.0551	5.47	0.165	Y
208	1.04	1.05	1.08	0.96	1.06	1.03	1.05	1.04	103.6	0.0355	3.43	0.107	Y
209	0.98	0.94	1.02	1.06	0.99	1.02	1.07	1.01	100.9	0.0441	4.38	0.132	Y

Attachment B

CS-PCB INITIAL MULTI LEVEL CALIBRATION

Column: GC15 XII 09/12/2006

Northeast Analytical Inc.

SDG No.: N/A

Instrument ID : GC 15

Initial Calibration Date(s): 09/27/2006

GC Column: CP-SIL5-C18

Calibration Standard File Names: ICAL0926A; ICAL0926B; ICAL0926C; ICAL0926D

Standard Concentration (1)		Internal Standard Concentration		I.S. Area	Standard Concentration (2)		Internal Standard Concentration		I.S. Area	Standard Concentration (3)		Internal Standard Concentration		I.S. Area:	Standard Concentration (4)		Internal Standard Concentration:		I.S. Area:	Calibration Curve	%RSD
(ng/ml):	0.1	(ng/ml):	18.18	183935	(ng/ml):	0.5	(ng/ml)	18.18	192051	(ng/ml):	5	(ng/ml):	182902		(ng/ml):	25	(ng/ml):	18.18	176149		
NEA ID #	RT	RRT	Peak Area	RF	NEA ID #	RT	RRT	Peak Area	RF	NEA ID #	RT	RRT	Peak Area	RF	NEA ID #	RT	RRT	Peak Area	RF		
1	14.494	0.11294		N/A	1	14.492	0.11295	111	47.76975	1	14.496	0.11297	1084	46.41980	1	14.496	0.11297	5315	45.57148	45.604	2.38
2	17.290	0.13473		N/A	2	17.286	0.13473		N/A	2	17.286	0.13471	150	334.43524	2	17.306	0.13486	741	326.83038	327.110	1.63
3	17.588	0.13706		N/A	3	17.594	0.13713	0	#DIV/0!	3	17.594	0.13711	596	84.46111	3	17.589	0.13707	3146	76.99193	77.232	6.54
4	17.893	0.13943	29	34.5163	4	17.888	0.13942	159	33.17347	4	17.899	0.13949	1664	30.23570	4	17.900	0.13950	8356	28.98869	29.034	8.05
5	18.124	0.14123	202	4.9965	5	18.128	0.14129	1099	4.80666	5	18.127	0.14127	10373	4.84927	5	18.129	0.14128	48514	4.99294	4.987	1.99
6	20.969	0.16340	202	5.0206	6	20.958	0.16335	1141	4.62966	6	20.957	0.16332	11225	4.48130	6	20.957	0.16331	51869	4.67006	4.662	4.86
7	21.193	0.16515	303	3.3402	7	21.196	0.16521	1655	3.19096	7	21.195	0.16518	16019	3.14030	7	21.198	0.16519	74163	3.26619	3.261	2.70
8	21.621	0.16848	164	6.1818	8	21.634	0.16861	927	5.69540	8	21.629	0.16856	8793	5.72066	8	21.631	0.16857	40867	5.92730	5.919	3.84
9	22.132	0.17246	317	3.1905	9	22.132	0.17250	1567	3.37097	9	22.124	0.17242	15344	3.27834	9	22.126	0.17243	71261	3.39920	3.394	2.86
10	22.360	0.17424	166	6.0860	10	22.358	0.17426	847	6.23959	10	22.355	0.17421	9730	5.17002	10	22.356	0.17422	45201	5.35898	5.351	9.24
11	22.854	0.17809	162	6.2263	11	22.862	0.17819	860	6.14374	11	22.855	0.17812	8951	5.61953	11	22.857	0.17812	41593	5.82379	5.815	4.74
12	25.322	0.19732	161	6.2877	12	25.316	0.19731	842	6.27145	12	25.314	0.19727	8107	6.20498	12	25.312	0.19725	37621	6.43866	6.429	1.57
13	25.989	0.20252	490	2.0632	13	25.980	0.20249	2649	1.99364	13	25.977	0.20244	24577	2.04672	13	25.979	0.20246	113894	2.12679	2.123	2.67
14	26.219	0.20431	198	5.1206	14	26.234	0.20447	1103	4.79076	14	26.227	0.20440	10975	4.58322	14	26.224	0.20437	51035	4.74638	4.740	4.68
15	26.897	0.20959	351	5.7569	15	26.900	0.20966	1862	5.67377	15	26.896	0.20960	17854	5.63495	15	26.902	0.20965	81010	5.98021	5.965	2.68
16	27.394	0.21347	355	2.8527	16	27.402	0.21357	2041	2.58830	16	27.390	0.21345	18638	2.69892	16	27.390	0.21345	86613	2.79668	2.793	4.25
17	27.604	0.21510	734	4.1361	17	27.640	0.21543	3769	4.20436	17	27.625	0.21529	34540	4.36914	17	27.632	0.21534	158154	4.59482	4.585	4.72
18	27.865	0.21714	312	3.2422	18	27.862	0.21716	1688	3.12871	18	27.860	0.21712	15687	3.20672	18	27.861	0.21712	72510	3.34064	3.335	2.72
19	28.271	0.22030	80	12.6684	19	28.250	0.22019	426	12.41090	19	28.239	0.22007	4128	12.18480	19	28.246	0.22012	19349	12.51920	12.506	1.64
20	28.502	0.22210	196	5.1622	20	28.522	0.22230	1063	4.97112	20	28.519	0.22225	10704	4.69945	20	28.522	0.22227	49166	4.92680	4.917	3.83
21	28.794	0.22438	349	2.8955	21	28.792	0.22441	2129	2.48046	21	28.792	0.22438	20056	2.50818	21	28.796	0.22441	92449	2.62014	2.615	7.22
22	31.454	0.24510	304	3.3253	22	31.469	0.24527	1736	3.04193	22	31.472	0.24527	16478	3.05277	22	31.476	0.24529	75627	3.20294	3.197	4.27
23	31.773	0.24759	449	2.2548	23	31.757	0.24752	2515	2.10034	23	31.756	0.24748	23989	2.09691	23	31.757	0.24748	110945	2.18332	2.180	3.49
24	32.460	0.25294	728	2.7810	24	32.464	0.25302	4206	2.51180	24	32.459	0.25296	38710	2.59897	24	32.449	0.25287	175812	2.75554	2.749	4.82
25	32.781	0.25545	367	2.7531	25	32.794	0.25560	2046	2.58218	25	32.781	0.25547	18809	2.67440	25	32.786	0.25550	85427	2.83551	2.828	4.00
26	32.977	0.25697	688	2.9395	26	32.979	0.25704	3857	2.73881	26	32.984	0.25705	36472	2.75843	26	32.984	0.25704	162064	2.98930	2.979	4.43
27	33.726	0.26281	392	2.5825	27	33.736	0.26294	1887	2.79955	27	33.725	0.26282	19596	2.56699	27	33.729	0.26284	89561	2.70461	2.699	4.12
28	34.397	0.26803	1446	2.7984	28	34.367	0.26786	8274	2.55365	28	34.360	0.26778	78315	2.56925	28	34.365	0.26780	339324	2.85544	2.842	5.76
29	34.583	0.26948	576	1.7551	29	34.598	0.26966	3099	1.70462	29	34.589	0.26956	29765	1.68999	29	34.592	0.26958	140152	1.72833	1.727	1.66
30	34.725	0.27059	341	2.9693	30	34.760	0.27093	1886	2.80045	30	34.755	0.27085	18727	2.68611	30	34.755	0.27084	86304	2.80669	2.802	4.14
31	35.099	0.27351	321	3.1475	31	35.068	0.27333	1789	2.95165	31	35.066	0.27328	17729	2.83732	31	35.063	0.27325	80283	3.01721	3.009	4.33
32	35.635	0.27768	473	2.1379	32	35.647	0.27784	2527	2.08985	32	35.641	0.27776	24971	2.01449	32	35.650	0.27781	116150	2.08549	2.083	2.44
33	38.455	0.29966	323	3.1328	33	38.446	0.29965	1774	2.97664	33	38.437	0.29955	17429	2.88618	33	38.444	0.29959	82026	2.95309	2.950	3.49
34	38.657	0.30123	451	2.2411	34	38.641	0.30117	2174	2.42915	34	38.631	0.30106	22105	2.27565	34	38.626	0.30101	100634	2.40702	2.401	4.01
35	38.818	0.30249	355	2.8500	35	38.838	0.30271	1962	2.69171	35	38.840	0.30268	21286	2.36314	35	38.834	0.30263	95095	2.54722	2.539	7.94
36	39.273	0.30603	521	1.9427	36	39.254	0.30595	2721	1.94128	36	39.255	0.30592	26688	1.88486	36	39.250	0.30587	122460	1.97802	1.974	1.99
37	39.508	0.30786	596	3.3933	37	39.504	0.30790	3253	3.24731	37	39.495	0.30779	34159	2.94525	37	39.501	0.30782	152538	3.17598	3.166	5.86
38	39.997	0.31167	391	2.5855	38	39.989	0.31167	2013	2.62400	38	39.981	0.31158	20607	2.44106	38	39.979	0.31156	91874	2.63653	2.628	3.49
39	40.076	0.31229	341	2.9629	39	40.130	0.31278	1807	2.92367	39	40.124	0.31270	17600	2.85812	39	40.128	0.31271	81163	2.98447	2.979	1.89
40	40.657	0.31682	1595	2.5375	40	40.642	0.31677	9579	2.20559	40	40.640	0.31672	87608	2.29672	40	40.650	0.31678	376058	2.57651	2.563	7.54
41	41.274	0.32162	1667	1.2138	41	41.257	0.32156	8942	1.18138	41	41.227	0.32129	81037	1.24149	41	41.214	0.32118	355561	1.36252	1.357	6.33
42	41.478	0.32321	636	3.1812	42	41.471	0.32323	3161	3.34223	42	41.467	0.32316	30313	3.31889	42	41.474	0.32320	143825	3.36838	3.366	2.53
43	41.788	0.32563	889	2.2762	43	41.781	0.32564	4858	2.17459	43	41.768	0.32551	42977	2.34091	43	41.781	0.32560	194843	2.48640	2.480	5.63
44	42.664	0.33246	514	1.9703	44	42.702	0.33282	3144	1.68022	44	42.689	0.33268	28664	1.75491	44	42.691	0.33268	131961	1.83561	1.832	6.86
45	43.022	0.33524	1180	2.5715	45	43.019	0.33530	6615	2.39554	45	43.007	0.33516	61977	2.43492	45	43.011	0.33518	272120	2.67047	2.660	5.02
46	43.621	0.33992	613	1.6498	46	43.591	0.33975	3015	1.75216	46	43.587	0.33968	31167	1.61396	46	43.586	0.33966	143283	1.69056	1.687	3.54
47	44.072	0.34343	253	3.9997	47	44.005	0.34298	1500	3.52126	47	44.010	0.34298	13014	3.86518	47	44.012	0.34299	59691	4.05803	4.050	6.23
48	46.067	0.35897	913	2.2161	48	46.042	0.35885	5054	2.09032	48	46.032	0.35873	48010	2.09551	48	46.035	0.35875	210364	2.30295	2.293	4.71
49	46.707	0.36396	1013	1.9977	49	46.759	0.36445	4703	2.24630	49	46.760	0.36441	46872	2.14639	49	46.768	0.36446	214613	2.25735	2.253	5.57
50	47.840	0.37279	506	1.9998	50	47.749	0.37216	2676	1.97346	50	47.752	0.37214	25179	1.99782	50	47.749	0.37210	119201	2.03210	2.031	1.20
51	47.928	0.37348	937	2.1601	51	47.921	0.37350	5405	1.95439	51	47.925	0.37349	51612	1.94929	51	47.927	0.37349	220429	2.19780	2.186	6.39

Attachment C Peak Identification Summary

NEA ID #	Identified Peaks RT	Surr Area: 284170 Surr % Rec: 80.34 Peak #	Peak Area	Total Peak Conc. (ng/mL)
1	14.489	1	3402	14.890039
2				0.000000
3	17.579	3	941	6.975080
4	17.891	4	12349	34.411436
5	18.121	5	3352	1.604376
6	20.949	6	2338	1.046155
7	21.193	7	1100	0.344268
8	21.618	8	13235	7.518167
9	22.097	9	181	0.058963
10	22.344	10	49159	25.248545
11	22.846	11	23067	12.874750
12				0.000000
13				0.000000
14	26.213	14	24354	11.078511
15	26.882	15	58555	33.523528
16	27.374	16	20232	5.422545
17	27.552	17	2482	1.092218
18	27.847	18	1127	0.360727
19	28.23	19	13979	16.778080
20	28.489	20	1330	0.627675
21	28.775	21	85844	21.548140
22	31.455	22	686	0.210458
23				0.000000
24	32.487	24	44603	11.766615
25	32.789	25	1008	0.273635

Attachment C Peak Identification Summary

26	32.969	26	53461	15.284243
27	33.711	27	66866	17.319559
28	34.24	28	51784	14.123876
29				0.000000
30	34.747	30	1281	0.344448
31	35.047	31	2745	0.792836
32	35.63	32	14582	2.914604
33	38.423	33	43439	12.300526
34	38.577	34	1073	0.247301
35	38.811	35	1016	0.247591
36	39.26	36	455	0.086206
37	39.465	37	58295	17.712125
38	39.955	38	1568	0.395459
39				0.000000
40	40.548	40	70423	17.322462
41	41.28	41	1663	0.216566
42	41.445	42	740	0.239089
43	41.77	43	11196	2.664826
44	42.676	44	635	0.111660
45	42.976	45	23956	6.114764
46	43.566	46	5414	0.876776
47	43.99	47	8389	3.260505
48	46.022	48	2728	0.600434
49	46.744	49	2195	0.474557
50				0.000000
51	47.895	51	6263	1.313792
52	48.451	52	1706	0.485703

Attachment C Peak Identification Summary

53	48.733	53	1112	0.179053
54	49.476	54	3519	0.605287
55	49.854	55	4226	0.710232
56				0.000000
57	50.407	57	30600	4.353927
58	50.543	58	3361	2.414252
59	51.116	59	25715	4.468041
60	51.556	60	715	0.127939
61	52.815	61	4637	0.776704
62				0.000000
63	53.378	63	3319	0.457120
64	54.214	64	3337	0.684454
65				0.000000
66	55.67	66	9089	1.905915
67	56.932	67	1142	0.218549
68	57.071	68	7586	1.550352
69				0.000000
70				0.000000
71	58.405	71	5675	0.969781
72				0.000000
73	58.892	73	4768	0.644404
74	59.495	74	1281	0.320384
75	60.318	75	6402	0.887570
76				0.000000
77	60.979	77	13011	1.854192
78	61.733	78	2491	0.432853
79				0.000000
80	63.645	80	5208	0.808123

Attachment C Peak Identification Summary

81				0.000000
82				0.000000
83	65.143	83	533	0.069225
84	66.037	84	4386	0.777548
85	66.74	85	678	0.113080
86	67.53	86	1869	0.290890
87	68.032	87	803	0.122388
88	68.789	88	2627	0.362221
89	68.857	89	1140	0.174226
90				0.000000
91	69.609	91	2743	0.406195
92	69.911	92	12746	2.002862
93				0.000000
94	70.857	94	1372	0.202063
95	71.318	95	1124	0.129263
96	71.673	96	526	0.081086
97	71.877	97	531	0.093696
98				0.000000
99	73.213	99	8025	1.101141
100				0.000000
101				0.000000
102	75.112	102	4985	0.781258
103				0.000000
104	75.804	104	1568	0.182421
105	76.943	105	1032	0.136377
106	77.561	106	5612	0.646922
107	77.673	107	3914	0.525489

Attachment C Peak Identification Summary

108				0.000000
109	78.644	109	8737	1.191774
110	79.317	110	3661	0.387708
111	79.83	111	2638	0.293613
112	80.248	112	1007	0.149980
113	81.961	113	1460	0.186808
114	82.239	114	2162	0.257639
115	82.686	115	2443	0.364268
116	83.41	116	1831	0.297039
117	84.33	117	1585	0.207644
118				0.000000
119	85.157	119	1879	0.244107
120	86.213	120	3408	0.387402
121	86.456	121	1202	0.165719
122	86.847	122	2362	0.385901
123	87.374	123	11244	1.217641
124	88.116	124	2304	0.281997
125	88.482	125	357	0.053163
126	89.348	126	6703	0.952080
127	91.17	127	8936	1.204686
128	91.662	128	2581	0.322760
129				0.000000
130	93.855	130	2271	0.260111
131				0.000000
132	95.708	132	6724	0.855547
133	96.733	133	1003	0.108451
134	99.342	134	6331	0.674417

Attachment C Peak Identification Summary

135	101.137	135	2129	0.223025
136	101.505	136	5573	0.704042
137				0.000000
138	103.348	138	1951	0.220482
139	103.98	139	1218	0.140854
140	107.227	140	3705	0.446574
141	107.947	141	995	0.104921
142	109.83	142		0.000000
143	115.852	143	2250	0.230500
144				0.000000
145	122.423	145	640	0.067503
146				0.000000
			Concentration	368.2597

Attachment D Peak Elution

	IUPAC#	Concentration (ng/mL)	IUPAC#	Concentration (ng/mL)	IUPAC#	Concentration (ng/mL)	IUPAC#	Concentration (ng/mL)
NEA ID # 1	1	14.890						
NEA ID # 2	2	0.000						
NEA ID # 3	3	6.975						
NEA ID # 4	4	38.125						
NEA ID # 5	10	1.604						
NEA ID # 6	9	1.046						
NEA ID # 7	7	0.344						
NEA ID # 8	6	7.518						
NEA ID # 9	5	0.059						
NEA ID # 10	8	27.587						
NEA ID # 11	19	12.875						
NEA ID # 12	14	0.000						
NEA ID # 13	30	0.000						
NEA ID # 14	18	11.079						
NEA ID # 15	11		17	35.98				
NEA ID # 16	27	5.423						
NEA ID # 17	12	0.131	13	0.55	24	0.42		
NEA ID # 18	16	0.361						

NEA ID # 19	*15*	**16.778**						
NEA ID # 20	*54*	**0.628**						
NEA ID # 21	*32*	**21.548**						
NEA ID # 22	*34*	**0.210**						
NEA ID # 23	*23*	**0.000**						
NEA ID # 24	*29*	**0.706**	*26*	**11.06**				
NEA ID # 25	*50*	**0.274**						
NEA ID # 26	*53*	**8.253**	*25*	**7.03**				
NEA ID # 27	*31*	**17.320**						
NEA ID # 28	*28*	**11.299**	*20*	**1.13**	*51*	**0.28**	*45*	**1.41**
NEA ID # 29	*21*	**0.000**						
NEA ID # 30	*33*	**0.344**						
NEA ID # 31	*46*	**0.793**						
NEA ID # 32	*22*	**2.915**						
NEA ID # 33	*52*	**12.301**						
NEA ID # 34	*73*	**0.247**						
NEA ID # 35	*43*	**0.248**						
NEA ID # 36	*69*	**0.086**						

NEA ID # 37	49	17.712	36					
NEA ID # 38	48	0.395						
NEA ID # 39	104	0.000						
NEA ID # 40	47	3.464	65		39		44	13.86
NEA ID # 41	75	0.217	62					
NEA ID # 42	96	0.014	59	0.22				
NEA ID # 43	38		42	2.66				
NEA ID # 44	41	0.112						
NEA ID # 45	35	0.245	40	2.51	71	3.36		
NEA ID # 46	64	0.877						
NEA ID # 47	37	3.261						
NEA ID # 48	103		72	0.60				
NEA ID # 49	68		94	0.47				
NEA ID # 50	57	0.000						
NEA ID # 51	100		95	1.31				
NEA ID # 52	93		58		102	0.49		
NEA ID # 53	98		67	0.18				
NEA ID # 54	63	0.605	88					

NEA ID # 55	*91*	**0.710**	*61*					
NEA ID # 56	*76*	**0.000**						
NEA ID # 57	*70*	**2.961**	*74*	**1.39**				
NEA ID # 58	*84*	**2.414**						
NEA ID # 59	*66*	**4.468**						
NEA ID # 60	*55*	**0.078**	*89*	**0.05**				
NEA ID # 61	*56*	**0.777**						
NEA ID # 62	*121*	**0.000**						
NEA ID # 63	*60*	**0.457**						
NEA ID # 64	*92*	**0.684**	*80*					
NEA ID # 65	*155*	**0.000**	*152*	**0.00**				
NEA ID # 66	*90*		*113*		*150*		*101*	**1.91**
NEA ID # 67	*136*	**0.129**	*83*	**0.09**				
NEA ID # 68	*99*	**1.550**						
NEA ID # 69	*145*	**0.000**						
NEA ID # 70	*112*	**0.000**						
NEA ID # 71	*119*	**0.029**	*97*	**0.92**	*86*	**0.02**	*109*	
NEA ID # 72	*125*	**0.000**						
NEA ID # 73	*87*	**0.644**						

NEA ID # 74	79	0.320				
NEA ID # 75	117	0.133	85	0.75	116	
NEA ID # 76	78	0.000				
NEA ID # 77	110	1.817	115	0.04		
NEA ID # 78	82	0.433	148			
NEA ID # 79	81	0.000				
NEA ID # 80	77	0.032	151	0.78	111	
NEA ID # 81	135	0.000	154	0.00		
NEA ID # 82	120	0.000				
NEA ID # 83	144	0.069				
NEA ID # 84	149	0.754	147	0.02		
NEA ID # 85	134	0.113	143			
NEA ID # 86	139	0.291	140			
NEA ID # 87	124	0.054	108		107	0.07
NEA ID # 88	131	0.362	142			
NEA ID # 89	123	0.174				
NEA ID # 90	106	0.000				
NEA ID # 91	132	0.406				
NEA ID # 92	118	2.003				

NEA ID # 93	*122*	**0.000**				
NEA ID # 94	*188*	**0.202**				
NEA ID # 95	*114*	**0.129**				
NEA ID # 96	*133*	**0.081**				
NEA ID # 97	*179*	**0.094**				
NEA ID # 98	*165*	**0.000**	*184*	**0.00**		
NEA ID # 99	*105*	**0.903**	*146*	**0.20**		
NEA ID # 100	*161*	**0.000**				
NEA ID # 101	*176*	**0.000**				
NEA ID # 102	*153*	**0.781**	*168*			
NEA ID # 103	*186*	**0.000**				
NEA ID # 104	*141*	**0.182**				
NEA ID # 105	*130*	**0.136**				
NEA ID # 106	*137*	**0.647**				
NEA ID # 107	*164*	**0.525**				
NEA ID # 108	*127*	**0.000**				
NEA ID # 109	*138*	**0.953**	*129*	**0.07**	*163*	**0.17**
NEA ID # 110	*160*	**0.388**				
NEA ID # 111	*158*	**0.294**				

NEA ID # 112	*178*	**0.150**				
NEA ID # 113	*175*	**0.187**				
NEA ID # 114	*128*	**0.250**	*166*	**0.01**		
NEA ID # 115	*187*	**0.364**				
NEA ID # 116	*182*	**0.297**	*126*			
NEA ID # 117	*183*	**0.208**				
NEA ID # 118	*185*	**0.000**				
NEA ID # 119	*174*	**0.244**				
NEA ID # 120	*159*	**0.387**				
NEA ID # 121	*177*	**0.166**				
NEA ID # 122	*202*	**0.386**	*162*			
NEA ID # 123	*181*	**1.218**				
NEA ID # 124	*171*	**0.262**	*173*	**0.02**		
NEA ID # 125	*167*	**0.053**				
NEA ID # 126	*201*	**0.952**				
NEA ID # 127	*204*	**1.205**				
NEA ID # 128	*156*	**0.274**	*157*	**0.01**	*197*	**0.04**
NEA ID # 129	*200*	**0.000**				
NEA ID # 130	*172*	**0.260**				

NEA ID # 131	*192*	**0.000**				
NEA ID # 132	*193*	**0.043**	*180*	**0.81**		
NEA ID # 133	*191*	**0.108**				
NEA ID # 134	*170*	**0.674**				
NEA ID # 135	*190*	**0.223**				
NEA ID # 136	*198*	**0.035**	*199*	**0.67**		
NEA ID # 137	*169*	**0.000**				
NEA ID # 138	*196*	**0.220**				
NEA ID # 139	*203*	**0.141**				
NEA ID # 140	*208*	**0.447**				
NEA ID # 141	*195*	**0.105**				
NEA ID # 142	*207*	**0.000**	*189*	**0.00**		
NEA ID # 143	*194*	**0.230**				
NEA ID # 144	*205*	**0.000**				
NEA ID # 145	*206*	**0.068**				
NEA ID # 146	*209*	**0.000**				
		285.21 376.77		69.54	4.84	**17.18**

Appendix E Sample Example

Company Name
Address
Address
Attn: Project Manager

Customer ID:	N/A	**NEA ID:**	Example
Sample Matrix:	SEDIMENT	**Date Sampled:**	N/A
Date Received:	N/A	**Time Sampled:**	N/A
Time Received:	N/A	**Date Extracted:**	N/A
Sampled By:	N/A	**Date Analyzed:**	09/28/2006
File ID:	N/A	**Date Printed:**	02/01/2007
Comment:	N/A	**NYS ELAP#: 11078**	

Method: Congener Specific PCB Analysis
PCB Data Summary Report

PCB HOMOLOG DISTRIBUTION

HOMOLOG	Weight %	Mole %
Mono	5.82	7.88
Di	24.95	28.56
Tri	38.13	37.80
Tetra	21.73	19.00
Penta	4.65	3.64
Hexa	2.13	1.51
Hepta	1.43	0.92
Octa	1.04	0.62
Nona	0.12	0.07
Deca	0.00	0.00

Ortho Cl/ Biphenyl Residue=	1.58
Meta Cl/ Biphenyl Residue=	0.64
Para Cl/ Biphenyl Residue=	0.72
Total Cl/ Biphenyl Residue=	2.94

TOTAL PCBs in SAMPLE= 82.8 mg/Kg

SURROGATE RECOVERY

Surrogate Analyte:	Congener IUPAC 207	
Amount Spiked:	0.0882	mg/Kg
Amount Recovered:	0.0708	mg/Kg
% Recovery:	80.3	%

Provided by: Northeast Analytical, Inc. 2190 Technology Drive, Schenectady, NY 12308
Phone: (518) 346-4592 Fax: (518) 381-6055

Appendix E Sample Example

Congener IUPAC #	CAS No.	No. of Cl's	Structure unprime-prime	Conc. mg/Kg	umoles/g	Total umoles	Weight %	Mole %	Qual.
001	2051-60-7	1	2-	3.282521	0.01739545	0.0986357	3.97	5.37	
002	2051-61-8	1	3-	ND	0.00000000	0.0000000	0.00	0.00	
003	2051-62-9	1	4-	1.537662	0.00814871	0.0462048	1.86	2.51	
004	13029-08-8	2	2-2	8.404576	0.03767179	0.2136066	10.15	11.62	
005	16605-91-7	2	23-	ND	0.00000000	0.0000000	0.00	0.00	
006	25569-80-6	2	2-3	1.657386	0.00742889	0.0421233	2.00	2.29	
007	33284-50-3	2	24-	0.075894	0.00034018	0.0019289	0.09	0.10	J
008	34883-43-7	2	2-4	6.081616	0.02725960	0.1545674	7.35	8.41	
009	34883-39-1	2	25-	0.230626	0.00103373	0.0058615	0.28	0.32	
010	33146-45-1	2	26-	0.353686	0.00158532	0.0089891	0.43	0.49	
011	2050-67-1	2	3-3	ND	0.00000000	0.0000000	0.00	0.00	
012	2974-92-7	2	34-	0.028894	0.00012951	0.0007343	0.03	0.04	J
013	2974-90-5	2	3-4	0.120390	0.00053962	0.0030598	0.15	0.17	
014	34883-41-5	2	35-	ND	0.00000000	0.0000000	0.00	0.00	
015	2050-68-2	2	4-4	3.698741	0.01657885	0.0940054	4.47	5.11	B
016	38444-78-9	3	23-2	0.079522	0.00030871	0.0017504	0.10	0.10	J
017	37680-66-3	3	24-2	7.931258	0.03078904	0.1745800	9.58	9.50	
018	37680-65-2	3	25-2	2.442266	0.00948085	0.0537583	2.95	2.92	
019	38444-73-4	3	26-2	2.838249	0.01101805	0.0624745	3.43	3.40	
020	38444-84-7	3	23-3	0.249090	0.00096696	0.0054829	0.30	0.30	
021	55702-46-0	3	234-	ND	0.00000000	0.0000000	0.00	0.00	
022	38444-85-8	3	23-4	0.642527	0.00249428	0.0141431	0.78	0.77	
023	55720-44-0	3	235-	ND	0.00000000	0.0000000	0.00	0.00	
024	55702-45-9	3	236-	0.091496	0.00035519	0.0020140	0.11	0.11	J
025	55712-37-3	3	24-3	1.549935	0.00601683	0.0341166	1.87	1.86	
026	38444-81-4	3	25-3	2.438322	0.00946553	0.0536715	2.95	2.92	
027	38444-76-7	3	26-3	1.195404	0.00464054	0.0263128	1.44	1.43	
028	7012-37-5	3	24-4	2.490896	0.00966963	0.0548287	3.01	2.98	
029	15862-07-4	3	245-	0.155638	0.00060418	0.0034258	0.19	0.19	
030	35693-92-6	3	246-	ND	0.00000000	0.0000000	0.00	0.00	
031	16606-02-3	3	25-4	3.818110	0.01482186	0.0840429	4.61	4.57	
032	38444-77-8	3	26-4	4.750304	0.01844062	0.1045620	5.74	5.69	
033	38444-86-9	3	34-2	0.075934	0.00029477	0.0016714	0.09	0.09	J
034	37680-68-5	3	35-2	0.046396	0.00018011	0.0010212	0.06	0.06	J
035	37680-69-6	3	34-3	0.053920	0.00020932	0.0011869	0.07	0.06	J
036	38444-87-0	3	35-3	ND	0.00000000	0.0000000	0.00	0.00	
037	38444-90-5	3	34-4	0.718781	0.00279030	0.0158216	0.87	0.86	
038	53555-66-1	3	345-	ND	0.00000000	0.0000000	0.00	0.00	
039	38444-88-1	3	35-4	ND	0.00000000	0.0000000	0.00	0.00	

Appendix E Sample Example

Congener IUPAC #	CAS No.	No. of Cl's	Structure unprime-prime	Conc. mg/Kg	umoles/g	Total umoles	Weight %	Mole %	Qual.
040	38444-93-8	4	23-23	0.552682	0.00189275	0.0107322	0.67	0.58	
041	52663-59-9	4	234-2	0.024616	0.00008430	0.0004780	0.03	0.03	J
042	36559-22-5	4	23-24	0.587463	0.00201186	0.0114076	0.71	0.62	
043	70362-46-8	4	235-2	0.054582	0.00018692	0.0010599	0.07	0.06	J
044	41464-39-5	4	23-25	3.055000	0.01046233	0.0593235	3.69	3.23	
045	70362-45-7	4	236-2	0.311362	0.00106631	0.0060462	0.38	0.33	
046	41464-47-5	4	23-26	0.174781	0.00059857	0.0033940	0.21	0.18	B
047	2437-79-8	4	24-24	0.763750	0.00261558	0.0148309	0.92	0.81	
048	70362-47-9	4	245-2	0.087179	0.00029856	0.0016929	0.11	0.09	J
049	41464-40-8	4	24-25	3.904652	0.01337209	0.0758225	4.72	4.13	
050	62796-65-0	4	246-2	0.060323	0.00020659	0.0011714	0.07	0.06	J
051	68194-04-7	4	24-26	0.062272	0.00021326	0.0012092	0.08	0.07	J
052	35693-99-3	4	25-25	2.711661	0.00928651	0.0526564	3.28	2.86	
053	41464-41-9	4	25-26	1.819489	0.00623113	0.0353317	2.20	1.92	
054	15968-05-5	4	26-26	0.138371	0.00047387	0.0026870	0.17	0.15	
055	74338-24-2	4	234-3	ND	0.00000000	0.0000000	0.00	0.00	
056	41464-43-1	4	23-34	0.171225	0.00058639	0.0033249	0.21	0.18	
057	70424-67-8	4	235-3	ND	0.00000000	0.0000000	0.00	0.00	
058	41464-49-7	4	23-35	ND	0.00000000	0.0000000	0.00	0.00	
059	74472-33-6	4	236-3	0.049545	0.00016967	0.0009621	0.06	0.05	J
060	33025-41-1	4	234-4	0.100772	0.00034511	0.0019568	0.12	0.11	J
061	33284-53-6	4	2345-	ND	0.00000000	0.0000000	0.00	0.00	
062	54230-22-7	4	2346-	ND	0.00000000	0.0000000	0.00	0.00	
063	74472-34-7	4	235-4	0.133436	0.00045697	0.0025911	0.16	0.14	
064	52663-58-8	4	236-4	0.193286	0.00066194	0.0037533	0.23	0.20	
065	33284-54-7	4	2356-	ND	0.00000000	0.0000000	0.00	0.00	
066	32598-10-0	4	24-34	0.984983	0.00337323	0.0191269	1.19	1.04	
067	73575-53-8	4	245-3	0.039472	0.00013518	0.0007665	0.05	0.04	J
068	73575-52-7	4	24-35	ND	0.00000000	0.0000000	0.00	0.00	
069	60233-24-1	4	246-3	ND	0.00000000	0.0000000	0.00	0.00	
070	32598-11-1	4	25-34	0.652682	0.00223521	0.0126741	0.79	0.69	
071	41464-46-4	4	26-34	0.741402	0.00253905	0.0143969	0.90	0.78	
072	41464-42-0	4	25-35	0.132366	0.00045331	0.0025703	0.16	0.14	
073	74338-23-1	4	26-35	0.054518	0.00018670	0.0010587	0.07	0.06	J
074	32690-93-0	4	245-4	0.307144	0.00105186	0.0059643	0.37	0.32	
075	32598-12-2	4	246-4	0.047742	0.00016350	0.0009271	0.06	0.05	J
076	70362-48-0	4	345-2	ND	0.00000000	0.0000000	0.00	0.00	
077	32598-13-3	4	34-34	ND	0.00000000	0.0000000	0.00	0.00	
078	70362-49-1	4	345-3	ND	0.00000000	0.0000000	0.00	0.00	
079	41464-48-6	4	34-35	0.070629	0.00024188	0.0013715	0.09	0.07	J
080	33284-52-5	4	35-35	ND	0.00000000	0.0000000	0.00	0.00	
081	70362-50-4	4	345-4	ND	0.00000000	0.0000000	0.00	0.00	
082	52663-62-4	5	234-23	0.095423	0.00029235	0.0016577	0.12	0.09	J
083	60145-20-2	5	235-23	ND	0.00000000	0.0000000	0.00	0.00	
084	52663-60-2	5	236-23	0.532224	0.00163059	0.0092458	0.64	0.50	

Appendix E Sample Example

Congener IUPAC #	CAS No.	No. of Cl's	Structure unprime-prime	Conc. mg/Kg	umoles/g	Total umoles	Weight %	Mole %	Qual.
085	65510-45-4	5	234-24	0.166316	0.00050955	0.0028892	0.20	0.16	
086	55312-69-1	5	2345-2	ND	0.00000000	0.0000000	0.00	0.00	
087	38380-02-8	5	234-25	0.142059	0.00043523	0.0024678	0.17	0.13	
088	55215-17-3	5	2346-2	ND	0.00000000	0.0000000	0.00	0.00	
089	73575-57-2	5	234-26	ND	0.00000000	0.0000000	0.00	0.00	
090	68194-07-0	5	235-24	ND	0.00000000	0.0000000	0.00	0.00	
091	68194-05-8	5	236-24	0.156571	0.00047969	0.0027199	0.19	0.15	
092	52663-61-3	5	235-25	0.150888	0.00046228	0.0026212	0.18	0.14	
093	73575-56-1	5	2356-2	ND	0.00000000	0.0000000	0.00	0.00	
094	73575-55-0	5	235-26	0.104617	0.00032052	0.0018174	0.13	0.10	J
095	38379-99-6	5	236-25	0.289627	0.00088734	0.0050314	0.35	0.27	
096	73575-54-9	5	236-26	ND	0.00000000	0.0000000	0.00	0.00	
097	41464-51-1	5	245-23	0.203100	0.00062224	0.0035282	0.25	0.19	
098	60233-25-2	5	246-23	ND	0.00000000	0.0000000	0.00	0.00	
099	38380-01-7	5	245-24	0.341776	0.00104711	0.0059373	0.41	0.32	
100	39485-83-1	5	246-24	ND	0.00000000	0.0000000	0.00	0.00	
101	37680-73-2	5	245-25	0.420160	0.00128726	0.0072990	0.51	0.40	
102	68194-06-9	5	245-26	0.107074	0.00032804	0.0018601	0.13	0.10	J
103	60145-21-3	5	246-25	ND	0.00000000	0.0000000	0.00	0.00	
104	56558-16-8	5	246-26	ND	0.00000000	0.0000000	0.00	0.00	
105	32598-14-4	5	234-34	0.199053	0.00060984	0.0034579	0.24	0.19	
106	70424-69-0	5	2345-3	ND	0.00000000	0.0000000	0.00	0.00	
107	70424-68-9	5	235-34	ND	0.00000000	0.0000000	0.00	0.00	
108	70362-41-3	5	234-35	ND	0.00000000	0.0000000	0.00	0.00	
109	74472-35-8	5	2346-3	ND	0.00000000	0.0000000	0.00	0.00	
110	38380-03-9	5	236-34	0.400583	0.00122728	0.0069589	0.48	0.38	
111	39635-32-0	5	235-35	ND	0.00000000	0.0000000	0.00	0.00	
112	74472-36-9	5	2356-3	ND	0.00000000	0.0000000	0.00	0.00	
113	68194-10-5	5	236-35	ND	0.00000000	0.0000000	0.00	0.00	
114	74472-37-0	5	2345-4	0.028496	0.00008730	0.0004950	0.03	0.03	J
115	74472-38-1	5	2346-4	ND	0.00000000	0.0000000	0.00	0.00	
116	18259-05-7	5	23456-	ND	0.00000000	0.0000000	0.00	0.00	
117	68194-11-6	5	2356-4	0.029350	0.00008992	0.0005099	0.04	0.03	J
118	31508-00-6	5	245-34	0.441533	0.00135273	0.0076703	0.53	0.42	
119	56558-17-9	5	246-34	ND	0.00000000	0.0000000	0.00	0.00	
120	68194-12-7	5	245-35	ND	0.00000000	0.0000000	0.00	0.00	
121	56558-18-0	5	246-35	ND	0.00000000	0.0000000	0.00	0.00	
122	76842-07-4	5	345-23	ND	0.00000000	0.0000000	0.00	0.00	
123	65510-44-3	5	345-24	0.038408	0.00011767	0.0006672	0.05	0.04	J
124	70424-70-3	5	345-25	ND	0.00000000	0.0000000	0.00	0.00	
125	74472-39-2	5	345-26	ND	0.00000000	0.0000000	0.00	0.00	
126	57465-28-8	5	345-34	ND	0.00000000	0.0000000	0.00	0.00	
127	39635-33-1	5	345-35	ND	0.00000000	0.0000000	0.00	0.00	
128	38380-07-3	6	234-234	0.055093	0.00015265	0.0008656	0.07	0.05	J
129	55215-18-4	6	2345-23	ND	0.00000000	0.0000000	0.00	0.00	

Appendix E Sample Example

Congener IUPAC #	CAS No.	No. of Cl's	Structure unprime-prime	Conc. mg/Kg	umoles/g	Total umoles	Weight %	Mole %	Qual.
175	40186-70-7	7	2346-235	0.041182	0.00010418	0.0005907	0.05	0.03	J
176	52663-65-7	7	2346-236	ND	0.00000000	0.0000000	0.00	0.00	
177	52663-70-4	7	2356-234	0.036533	0.00009242	0.0005240	0.04	0.03	J
178	52663-67-9	7	2356-235	0.033063	0.00008364	0.0004743	0.04	0.03	J
179	52663-64-6	7	2356-236	ND	0.00000000	0.0000000	0.00	0.00	
180	35065-29-3	7	2345-245	0.179176	0.00045327	0.0025701	0.22	0.14	
181	74472-47-2	7	23456-24	0.268430	0.00067905	0.0038504	0.32	0.21	
182	60145-23-5	7	2345-246	0.065483	0.00016565	0.0009393	0.08	0.05	J
183	52663-69-1	7	2346-245	0.045775	0.00011580	0.0006566	0.06	0.04	J
184	74472-48-3	7	2346-246	ND	0.00000000	0.0000000	0.00	0.00	
185	52712-05-7	7	23456-25	ND	0.00000000	0.0000000	0.00	0.00	
186	74472-49-4	7	23456-26	ND	0.00000000	0.0000000	0.00	0.00	
187	52663-68-0	7	2356-245	0.080303	0.00020315	0.0011519	0.10	0.06	J
188	74487-85-7	7	2356-246	0.044545	0.00011269	0.0006390	0.05	0.03	J
189	39635-31-9	7	2345-345	ND	0.00000000	0.0000000	0.00	0.00	
190	41411-64-7	7	23456-34	0.049166	0.00012438	0.0007052	0.06	0.04	J
191	74472-50-7	7	2346-345	0.023908	0.00006048	0.0003429	0.03	0.02	J
192	74472-51-8	7	23456-35	ND	0.00000000	0.0000000	0.00	0.00	
193	69782-91-8	7	2356-345	ND	0.00000000	0.0000000	0.00	0.00	
194	35694-08-7	8	2345-2345	0.050814	0.00011823	0.0006704	0.06	0.04	J
195	52663-78-2	8	23456-234	0.023130	0.00005382	0.0003051	0.03	0.02	J
196	42740-50-1	8	2345-2346	0.048605	0.00011309	0.0006412	0.06	0.03	J
197	33091-17-7	8	2346-2346	ND	0.00000000	0.0000000	0.00	0.00	
198	68194-17-2	8	23456-235	ND	0.00000000	0.0000000	0.00	0.00	
199	52663-75-9	8	2345-2356	0.147446	0.00034306	0.0019452	0.18	0.11	
200	52663-73-7	8	23456-236	ND	0.00000000	0.0000000	0.00	0.00	
201	40186-71-8	8	2346-2356	0.209887	0.00048834	0.0027690	0.25	0.15	
202	2136-99-4	8	2356-2356	0.085072	0.00019793	0.0011223	0.10	0.06	J
203	52663-76-0	8	23456-245	0.031051	0.00007225	0.0004096	0.04	0.02	J
204	74472-52-9	8	23456-246	0.265574	0.00061790	0.0035036	0.32	0.19	
205	74472-53-0	8	23456-345	ND	0.00000000	0.0000000	0.00	0.00	
206	40186-72-9	9	23456-2345	ND	0.00000000	0.0000000	0.00	0.00	
207	52663-79-3	9	23456-2346	ND	0.00000000	0.0000000	0.00	0.00	
208	52663-77-1	9	23456-2356	0.098448	0.00021208	0.0012025	0.12	0.07	J
209	2051-24-3	10	23456-23456	ND	0.00000000	0.0000000	0.00	0.00	

Appendix E Sample Example

Total PCB Conc. =	82.8 mg/Kg	CP-SIL5-C18 Internal Std Retention Time =	128.284	minutes
Total umoles/g =	0.3242	CP-SIL5-C18 Internal Std Peak Area =	189423	
Total umoles =	1.8380	Corrected MDL:	0.022045	mg/Kg
		Corrected PQL:	0.11023	mg/Kg

Notes:

(1) J - Qualifier denotes concentration reported is less than the Practical Quantitaion Limit (PQL) but greater than the Method Detection Limit (MDL)
B- Qualifier indicates an estimated value. IUPAC 15 was detected at 0.003158mg/kg, IUPAC46 was detected at 0.000587mg/kg, in the associated blank (AJ12196B) at a level exceeding the Practical Quantitation Limit (PQL) 0.001266mg/kg

(2) ND -Denotes analyte not detected (concentration less than the MDL)

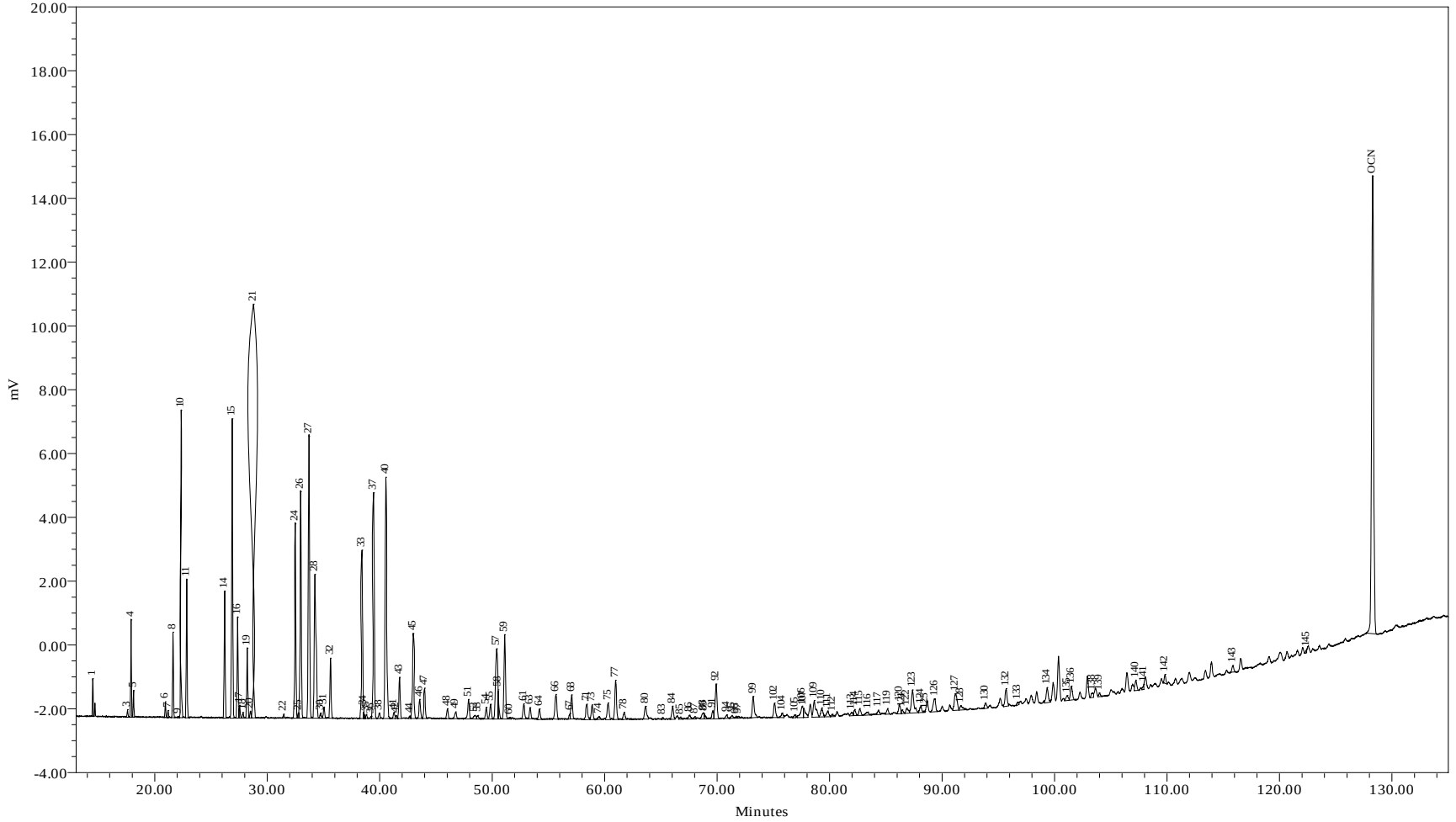
(3) PQL and MDL for IUPAC #s 1, 2, and 3 are 5 times higher than all other Congeners

(4) The above listed IUPAC numbers and congener names are consistent with the CAS numbering system. Note that G. Frame's 1996 PCB publication used Guitart's proposed renumbering for congeners 107,108, and 109, which has not to date been adopted by CAS, Ballschmiter or IUPAC.

IUPAC# & BZ#	Guitart et al.
107	109
108	107
109	108

Appendix E Sample Example

Chromatogram Report Comprehensive Quantitative Congener Specific PCB
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518) 346-4592 Fax: (518) 381-6055



NORTHEAST ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

NE142_07.SOP
REVISION NUMBER: 07

STANDARD OPERATING PROCEDURE FOR EXTRACTION AND PREPARATION OF AQUEOUS SAMPES BY SW-846 METHOD 3520C FOR SUBSEQUENT ANALYSIS BY EPA METHOD 8082 COMPREHENSIVE QUANTITATIVE CONGENER SPECIFIC ANALYSIS

March 13, 2007

COPY #

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STANDARD OPERATING PROCEDURE

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1.0 Identification of Test Methods

Standard Operating Procedure for the Extraction and Cleanup of Aqueous Samples using the Continuous Liquid/Liquid Extraction Technique (EPA SW-846 Method 3520C for Subsequent Analysis by EPA/NEA-Comprehensive Quantitative Congener Specific PCB Analysis (EPA 8082-CQCS)

2.0 Applicable Matrix or Matrices

This method is applicable to surface water, groundwater, wastewater and other aqueous samples.

3.0 Detection Limits

Please see consult determinative method (EPA Method 8082 Comprehensive Quantitative Congener Specific Analysis (CQCS) Lab SOP # NE133.doc) for details.

4.0 Scope and Application

The following procedure is utilized by Northeast Analytical, Inc for the extraction, extract concentration and subsequent extract cleanup of PCBs in aqueous samples using the continuous liquid-liquid extraction technique.

5.0 Summary

5.1 A measured volume of sample (usually 1 liter) is adjusted to appropriate pH and placed into a continuous liquid liquid extractor then extracted with Dichlolmethane for 2 hours.

5.2 The extract is concentrated, exchanged to Hexane and set to final volume. Default set volume is 10mL. Client and/or project specifications may dictate alternative set volume.

5.3 The sample extract is cleaned up using appropriate cleanup steps (acid wash, florisil, elemental sulfur removal with Mercury) and then presented for GC analysis for PCB Congeners.

6.0 Definitions

Matrix Spike:
An aliquot of sample, fortified with a known quantity of the analytes of interest, used to indicate the accuracy of the method by measuring recovery.

Surrogate Compound Spike:
In chemical composition and chromatography similar to the analytes of interest. Usually not found in environmental samples. These compounds are spiked into all samples, standards, blanks, and matrix spike samples prior to analysis. Percent recoveries are calculated for each surrogate.

QC- Quality Control:
A set of measures for each sample within an analysis methodology to assure that the process is in control.

Internal Standard:
A pure compound added to a sample just before instrumental analysis to permit corrections for inefficiencies.

7.0 Interferences

Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of certain plastics. Phthalate esters respond on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Samples and extracts should not be exposed to plastics such as gloves, tubing, coating on clamps, and pipette bulbs, etc.

8.0 Safety

The extraction chemist should have received in-house safety training and should know the location of first aid equipment and emergency spill / clean-up equipment, before handling any apparatus or equipment. Safety glasses and gloves must be worn when handling glassware and samples. Polychlorinated biphenyls (PCBs) have been tentatively classified as known or suspected carcinogens. The extraction chemist must review the Material Safety Data Sheet (MSDS) for PCBs and all reagents used in the procedure before beginning the extractions. All equipment and solvents should be handled within a lab fume hood.

9.0 Equipment and Supplies

9.1 **Accelerated One-Step Extractor/Concentrator***;* Corning #3915-C Consisting of:

a) **Jacketed Concentrator Tube**; Corning #2157-100TJ
b) **Membrane Assembly, 47 mL**; Corning #3918-47

c) **Extractor Body**; Corning #3915-MBO
d) **Extractor Cup;** Corning #3915-ECO
e) **Snyder Sidearm;** Corning #3915-MSCO
f) **Coupler Assembly;** Corning #3915-CAO
g) **Photoceram Membrane Support;** Corning #3918-47MS
h) **Stopcock Assembly;** Corning #3915-SCA
i) **Connectors/Washers for Stopcock Assembly;** Corning #3915-CW
j) **35/25 Joint O-Ring, Viton;** Corning #3915-35VO
k) **Coupler O-Ring;** Corning #3915-CVO
l) **35/25 Joint Clamp;** Corning #2140-35
m) **Hose Connectors for Jacketed Concentrator Tube** Corning #2158-CO
n) **24/40 Taper Joint Clamp**
o) **Stainless Steel Ball-Joint Clamp**

9.2 **Allihn Condenser**; medium; Corning #3480-MCO

9.3 **Constant Temperature Circulator**; VWR Scientific and Lauda (one each)

9.4 **Neslab Chiller**; CFT-33

9.5 **Graduated Cylinder**; 100mL; Kimble #20011-100

9.6 **Beakers**, assorted Pyrex 600mL and 1000mL, used for liquid containment and pipette storage.

9.7 **Glass Wool**; Silanized, Supleco #2-0410, solvent washed as per NE153.doc

9.8 **Glass Beads**

9.9 **Disposable Pasteur 9'' pipettes**; Krackeler #67-450-900-CS or equivalent

9.10 **pH Indicator Strips**; pH Range 0-14, Sensitivity 1.0, EM Science # 9590

9.11 **Microliter Gastight Syringe** – two 1000 microliter; Hamilton

9.12 **TurboVap Evaporator concentrator tubes** (Turbo Tubes); Zymark 250mL, 0.5mL endpoint.

9.13 **TurboVap Evaporator**; Zymark #ZW640-3

9.14 **10mL Volumetric**; Pyrex #5640

9.15 **40mL and 4-dram vials** with polyseal cap for sample extract

9.16 **Vial rack**; plastic rack used to hold vials during all phases of the extract processing.

9.17 **Centrifuge**; International Equipment Co., Model CL (or equivalent)

9.18 **Wrist Shaker**; Burrel wrist action shaker, Model 75 and 88 (or equivalent)

9.19 **Auto Sampler vials**; Scientific Resources Inc. #99468-A

10.0 Reagents and Standards

10.1 **1:1 Sulfuric acid** (H2SO4)
Preparation: To a beaker containing 500 mL cold DI-water slowly and under constant stirring add 500 mL concentrated H2SO4. Allow the mixture to cool after preparation.)

10.2 **5N Sodium Hydroxide,** J.T.Baker #5761-03, UN1824

10.3 **Dichloromethane**; High purity solvent Baxter (Burdick\Jackson), UN1593

10.4 **Hexane**, High purity solvent Baxter; (Burdick\Jackson) UN1208

10.5 **Acetone**; High purity solvent Baxter; (Burdick\Jackson) UN1090

10.6 **Concentrated Sulfuric Acid,** Mallinckrodt #2468, UN1830, Solvent washed as per NE174.doc

10.7 **Florisil**; J.T.Baker #M368-08, solvent washed per NE094.doc
SEE SUPERVISOR FOR THE APPROPRIATE FLORISIL DEACTIVATION CONCENTRATION TO BE USED.

10.8 **Mercury**; Triple distilled, Mercury Waste Solutions Inc., Solvent washed as per NE175.doc (or equivalent)

11.0 Sample collection, Preservation, Shipment and Storage

The samples should be collected in unpreserved 1 liter glass bottles with Teflon lined cap. Samples should be delivered to the laboratory on ice and should be chilled to 4± 2 Degrees C. Samples are stored refrigerated at the laboratory at 4 ± 2 degrees C. The sample extraction hold time for this method is five days from the date the samples were collected (not the usual seven days).

12.0 Quality Control

12.1 The extraction chemist should have completed an acceptable demonstration of precision and accuracy before performing the method without supervision. The addition of spiking material to a sample or blank must be witnessed by another extraction chemist and signed in the extraction logbook. All surrogates and matrix spikes must meet acceptable QC limits. An approved instructor must also certify the extraction chemist to perform the procedure.

12.2 A Method Blank sample and Lab Control Spike (LCS) must be prepared per each extraction batch or 1 per 20 site samples whichever is more frequent. A matrix spike/matrix spike duplicate (or lab duplicate) should be prepared for every 20 site samples or as per client specified quality assurance project plan (QAPP). Spike default for LCS, MS, MSD is 1.0 mL of A1242 @ 1.0ppm. Client and/or project specifications may dictate and alternate spike amount or Aroclor.

12.3 PCB Surrogate Nonachlorobiphenyl is added to each sample prior to extraction to measure extraction/cleanup efficiency. Default surrogate is: 0.5mL of 5'NCBP @ 0.2ppm. Client and/or project specifications may dictate an alternate surrogate concentration.

13.0 Calibration and Standardization

Please see consult determinative method (EPA Method 8082 Comprehensive Quantitative Congener Specific Analysis (CQCS) Lab SOP # NE133.doc) for Calibration and Standardization.

14.0 Procedure

Note: Unless otherwise noted, when rinsing is referred to in this SOP, it will be with a Teflon squirt bottle. A solvent followed by a (b) will be used to designate solvent from a beaker.

14.1 SAMPLE PREPARATION

14.1.1 Throughout the entire process it should be noted that if the extraction chemist encounters any problems or difficulties with any samples or steps involved, all work should **STOP!!!** Any problems should be brought to the attention of the supervisor and documented in the sample extraction logbook.

14.1.2 Before any steps are taken; the extraction chemist should first review the sample job folder and check the sample labels versus the original chain of custody. Any discrepancies should be noted in the sample extraction logbook.

14.1.3 Bring samples to room temperature by letting the samples warm up in the laboratory for a minimum of 30 minutes.

14.1.4 Mark the level of the sample on the outside of the sample container with a felt tip pen. Determine the pH of the sample by removing a small amount of sample with a Pasteur pipette (approximately 0.1 mL) and wet a pH indicator strip. The pH should be between 5 and 9. Adjust the pH with 1:1 Sulfuric Acid if the pH is greater than 9 or 5N Sodium Hydroxide if the pH is lass than 5. Record the original pH in the extraction logbook.

14.1.5 Using 1 Liter clear sample bottles, measure out two, 1 Liter aliquots of DI water to be used as an extraction blank and laboratory control spike.

14.2 ASSEMBLY OF CORNING ACCELERATED ONE STEP EXTRACTOR/CONCENTRATOR

See NE233.SOP

14.3 EXTRACTION

14.3.1 **Observe sample:** In case of a high amount of sediment or very fine particles place glass wool and some glass beads on top of membrane assembly. This will prevent the obstruction of the membrane. **DO NOT** pack glass wool down, otherwise you will obstruct the flow through the membrane.

14.3.2 Pour 55mL Dichloromethane (b) into extractor body. Check extractor for leaks, which will most likely occur between the extractor body and cup assembly.

14..3.3 Add the sample to the extractor unit. Rinse sample bottle with 55mL of Dichloromethane (b) and add to extractor unit.

14.3.4 Spike surrogate- and matrix-spike compound solutions directly into sample in extractor body.

14.3.5 Place condenser into top of extractor body

14.3.6 Open stopcock when both, the chiller and constant temperature circulator have reached proper operating temperature. Solvent will begin to flow from extractor body into concentrator tube and immediately begin to boil.

14.3.7 Periodically verify that the boiling rate of the solvent is approximately 900mL per hour (once the unit is running and stabilized) as follows:

a) Note solvent level in concentrator tube

b) Close stopcock and record how long it takes to boil 50mL of solvent over into extractor body.

c) The solvent should boil off in about 3 to 3.5 minutes, if the boiling rate is correct.

d) If **boiling rate** is too **slow**: Increase the temperature of the hot water circulator bath.
If **boiling rate** is too **fast**: Decrease the temperature of the hot water circulator bath.

14.3.8 Allow extraction to proceed for 2 hours.

14.3.9 Fill sample bottle with water to previous level. Pour water into a 1 Liter graduated cylinder and record the sample volume in the extraction logbook. Discard sample container according to guidelines in the Chemical Hygiene Plan.

14.4 SAMPLE EXTRACT CONCENTRATION

14.4.1 Close stopcock and allow solvent in concentrator tube to boil down.

14.4.2 Disconnect the concentrator tube from the hot water flow, when desired concentration volume of less than 10mL is reached. (**Note:** Sample will continue to boil, while apparatus is cooling down. Sample extract will reflux from Snyder sidearm, which will increase the extract volume.)

14.3.4 Attach the cold-water tubing to the concentrator tube.

14.3.5 Start cold water flow and cool an additional 10 to 15 minutes. (**Note:** make sure extract is cold before disconnecting concentrator tube or loss of analytes may occur.)

14.5 DISASSEMBLY

See NE233.SOP

14.6 TRANSFER OF SAMPLE

14.6.1 Using a Pasteur pipette transfer the sample extract into a correctly labeled and Hexane pre-rinsed TurboTube or 40mL vial.

14.6.2 Rinse inside of concentrator tube with 3 Pasteur pipette volumes of Hexane and add to the sample extract. Repeat this step twice more. Rinse down the inside walls of the Turbo Tube. Add enough Hexane to the extract to get a total volume of approximately 100 mL in the TurboTube OR in the case of the 40mL vial add enough Hexane for a total of approximately 20 mL.

14.7 SOLVENT REDUCTION: TURBOVAP EVAPORATOR SYSTEM

14.7.1 The TurboVap evaporator system is used to reduce the sample volume. The TurboVap uses a heated water bath and positive pressure nitrogen flow \ vortex action. The unit maintains a slight equilibrium imbalance between the liquid and the gaseous phase of the solvent extract, which allows fractional reduction of the solvent without loss of higher boiling point analytes.

14.7.2 Turn the unit on and allow to heat up to 40°C ± 2°C.

14.7.3 As a precaution the TurboVap system regulators should be checked to assure that there is no residual gas pressure within the system and that the gas pressure regulator is off before placing samples in the apparatus. Residual gas pressure may cause splashing and therefore cause cross contamination of samples. To bleed the system of residual gas pressure, place an empty TurboTube into water bath and close lid. Make sure that the nitrogen gas pressure regulator is closed. Bleed any residual gas until the regulator gauge reads "0" psi. Remove empty TurboTube.

14.7.4 If the sample was initially stored in a 40mL vial, pour the sample into a correctly labeled and Hexane pre-rinsed TurboTube. To complete the transfer, rinse the threads with one Pasteur pipette volume of Hexane. Rinse the 40mL vial with three Pastuer pipette volumes of Hexane. Add this to the TurboTube. Repeat this twice more. Rinse down the inside walls of the TurboTube. Bring the volume up to approximately 100 mL with Hexane.

14.7.5 Wipe down the inside of TurboVap with Hexane wetted paper towel. Place TurboTubes containing the sample extract into TurboVap and close lid. Slowly open the pressure regulator. Keep the gas pressure very low to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the volume reduction.

14.7.6 The process for solvent (Dichloromethane \ Hexane) reduction takes approximately 30 minutes. **DO NOT** leave the unit unattended as extracts may be blown to dryness and PCB loss may occur. Immediately notify the extraction supervisor if an extract is blown to dryness and note the incident in the extraction logbook.

14.7.7 Concentrate the solvent to approximately 5.0mL. Remove the samples from the TurboVap and place into tube rack.

14.7.8 Using a Pasteur pipette quantitatively transfer the sample extract into a Hexane pre-rinsed 10 mL volumetric. Use Hexane to rinse the TurboTube and set to volume. Invert the volumetric several times to mix completely.

14.7.9 Transfer sample from the volumetric to a correctly labeled 4-dram vial.

14.8 SAMPLE EXTRACT CLEAN-UP PROCEDURE

Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection contain co-extracted xenobiotics and other interfering substances, which must be removed before accurate chromatographic analysis can be performed. Not all clean-up procedures need to be performed on every sample and several are sample matrix specific. The experience of the analyst combined with the sampling site history should guide the selection of which clean-up procedures are necessary. The extraction chemist records the sequence and number of repeats of cleanup steps performed in the sample logbook. Sample extract cleanups are performed on set volume extracts. The set volume is 5mL or 10mL for water/wastewater samples.

14.8.1 Sulfuric Acid Wash

14.8.1.1 The concentrated sulfuric acid treatment removes hydrocarbons and other organic compounds, which are Co-extracted with the PCB residues.

14.8.1.2 Add one Pasteur pipette full of solvent washed concentrated Sulfuric Acid (NE174.doc) to each extract vial and shake by hand, in a fume hood for 30 seconds. Then centrifuge for at least 1 minute and setting #4. Transfer the Hexane (upper) layer to a correctly labeled, pre-rinsed 4dram vial.

14.8.1.3 Repeat 14.8.1.2 if the sample extract appears to be heavily loaded (opaque) with colored material. Two to three acid washes may be required. **Note:** It is entirely possible that all colored material will not be removed from the extract.

14.8.2 Florisil Clean-Up

14.8.2.1 The Florisil removes co-extracted polar compounds, residual water and residual sulfuric acid. **SEE SUPERVISOR FOR THE APPROPRIATE FLORISIL DEACTIVATION CONCENTRATION TO BE USED.**

14.8.2.2 Add one spatula of Florisil to each extract vial and shake by hand for 30 seconds. Swirl to get any florisil off the walls of the vial, then allow to settle. Transfer the Hexane (upper) layer to a correctly labeled, pre-rinsed 4dram vial.

14.8.3 Removal of Sulfur Using Mercury

NOTE: Mercury is a highly toxic metal; all operations involving Mercury should be performed within a fume hood. Prior to using Mercury, the extraction chemist should become acquainted with proper handling and emergency spill/clean-up procedures associated with this metal and must have reviewed the Material Safety Data Sheet (MSDS).

14.8.3.2 Add 1-3 drops of solvent washed Mercury (NE175.doc) to the sample extracts, and cap. Handshake for 15-30 seconds. If the Mercury changes color or breaks up into tiny balls and will not reform the original ball, change the Mercury. To change the Mercury transfer extract into a new correctly labeled and Hexane pre-rinsed 4-dram vial and add new Mercury to it. Repeat previous step. Place on wrist shaker for 30 minutes. The sulfur is converted to

Mercuric Sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental Sulfur.

14.8.3.3 Remove the sample extracts from the wrist shaker.

14.9 FINAL EXTRAT PREPARATION

14.9.1 Transfer the extract to a Hexane pre-rinsed and correctly labeled final 4-dram vial. Labeled wth the client, the test, the dilution factor, the date, and the sample ID number.

14.9.2 The analyst will add Internal Standard before analysis. For the Internal Standard spiking procedures refer to the analyst.

14.9.3 Submit the final vials along with the project folder containing the Extraction Log sheet and GCQueue Lab Sheet to the GC analyst.

15.0 Calculations

Please see consult determinative method (EPA Method 8082 Comprehensive Quantitative Congener Specific Analysis (CQCS) Lab SOP # NE133.doc) for details.

16.0 Method Performance

Please consult determinative (EPA Method 8082 Comprehensive Quantitative Congener Specific Analysis (CQCS) Lab SOP # NE133.doc) for Method Performance details.

17.0 Pollution Prevention

See NEA168.SOP

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Please see consult determinative method (EPA Method 8082 Comprehensive Quantitative Congener Specific Analysis (CQCS) Lab SOP # NE133.doc) for Data Assessment and Acceptance Criteria for Quality Control Measures.

19.0 Corrective Action for Out-Of-Control Data

Please see consult determinative method (EPA Method 8082 Comprehensive Quantitative Congener Specific Analysis (CQCS) Lab SOP # NE133.doc) for Corrective Action for Out-Of-Control Data

20.0 Contingencies for Handling Out-Of-Control or Unacceptable Data

Please see consult determinative method (EPA Method 8082 Comprehensive Quantitative Congener Specific Analysis (CQCS) Lab SOP # NE133.doc) for or Contingencies for Handling Out-Of-Control or Unacceptable Data

21.0 Waste Management

See NEA054.SOP, NEA083.SOP, NEA089.SOP

22.0 REFERENCES

1. U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

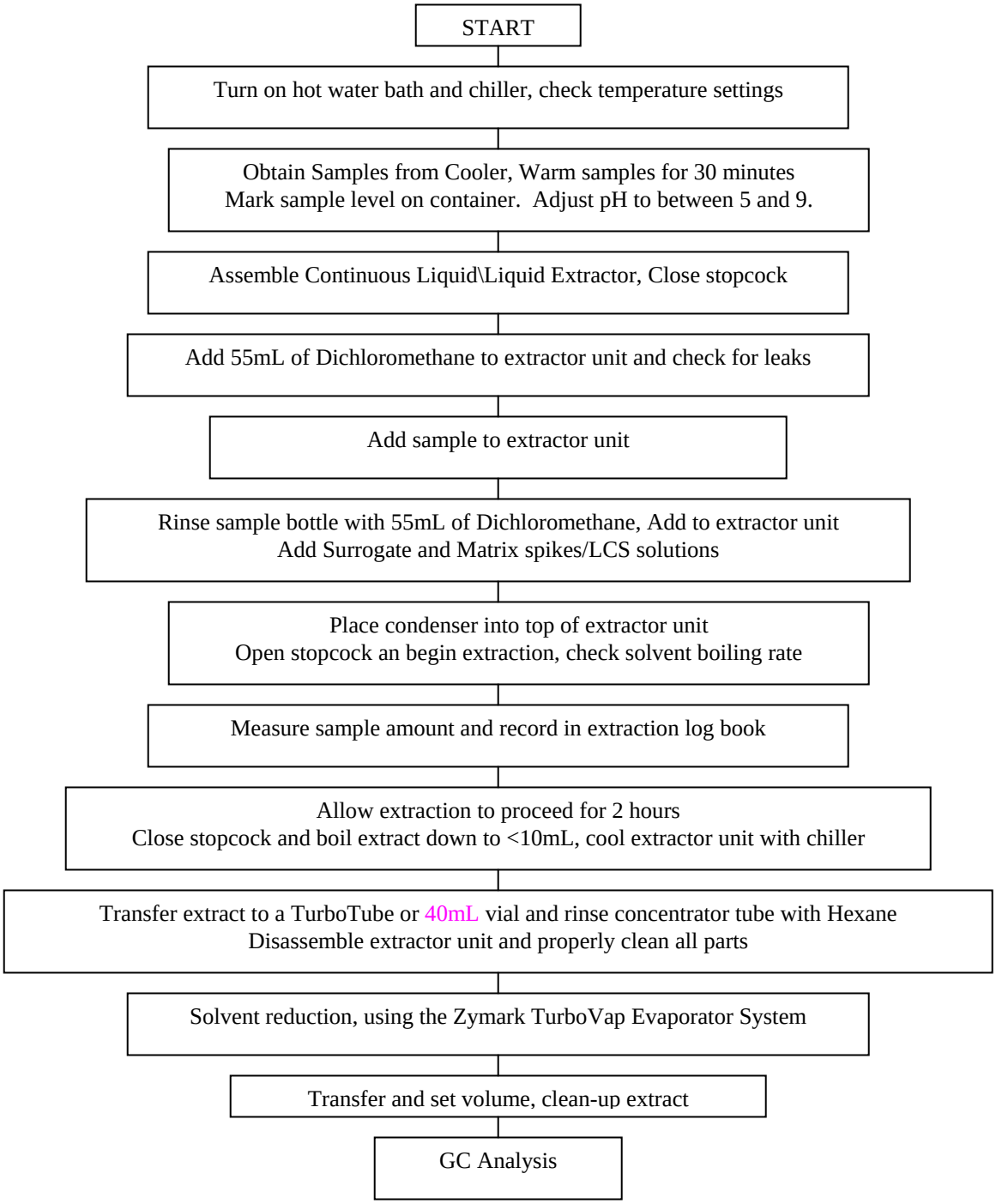
2. "Guide to Environmental Analytical Methods", Fourth Edition, Genium Publishing Corporation, 1998.

3. Northeast Analytical Inc. Laboratory SOP NE133.doc EPA Method 8082 with Comprehensive Quantitative Congener Specific Analysis.

23.0 Tables, Diagrams, Flowcharts and Validation Data

23.1Method Flowchart

Attachment 1



STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

NE143_04.DOC
REVISION NUMBER: 04

Standard Operating Procedures for the Extraction and Cleanup of Soil, Sediment, and Solid Samples for Polychlorinated Biphenyl (PCB) Analysis Using the ASE Extraction Technique (SW-846 method 3545) and Subsequent Analysis by SW-846 Method 8082
December 28, 2006

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STANDARD OPERATING PROCEDURE

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Revision: 04

NELAP Annual Review:
Date last reviewed:
Reviewed by:

1.0 TITLE/PURPOSE

Title:

Standard Operating Procedure for the extraction and cleanup of soil, sediment, and solid samples for Polychlorinated Biphenyl (PCB) analysis using the ASE extraction technique by SW-846 Method 3545 and subsequent Analysis by SW-846 Method 8082.

Purpose:

The purpose of this SOP is to provide to the chemist the procedures required to perform extractions of PCBs, in soil/sediment/solid sample, using the ASE extraction technique and to perform the subsequent extract volume reduction and cleanup .

2.0 APPLICABLE MATRICES

This test method is appropriate for Soil, sediment, and solid samples. Other extraction methods such as the soxhlet or sonication technique may be used in place of the ASE extraction at the discretion of the supervising chemist. Extract cleanup steps employed may vary from sample to sample, matrix to matrix. The chemist must have an understanding of the methods and requirements of USEPA-SW- 846A "Test Methods for Solid Wastes" Volume 1B: Lab Manual, 3rd edition. Methods 3545, 3500, 2500A. An approved instructor must also certify the chemist to perform the procedure. In general, samples designated as high level may have expected concentrations > 10 ug/L (10 PPB).

3.0 DETECTION LIMIT

Please see determinative method (Lab SOP NE148) for details.

4.0 SCOPE AND APPLICATION

The following procedure is utilized by Northeast Analytical, Inc. for the extraction and cleanup of PCBs from soil/sediment/solid samples using the ASE extraction method for subsequent analysis by SW-846 Method 8082.

5.0 SUMMARY OF TEST METHOD

The extraction and cleanup of soil, sediment, and solid samples for Polychlorinated Biphenyl (PCB) analysis using the ASE extraction technique SW-846 method 3545 begins with the weighing out of 10-11 grams of the sample. Next the sample is dried using hydromatrix and loaded into individual ASE cells where they are spiked and surrogated. Then utilizing the proper method and 1:1 Hexane and Acetone the sample is extracted via the Accelerated Solvent Extractor. The extracted solvent then undergoes several cleaning steps including the removal of water and exposure to acid, TBA, florisil, and mercury. Then the cleaned extract is properly diluted and sent for GC analysis.

6.0 DEFINITIONS

Blank: A non site sample created in the laboratory to evaluate cross contamination of the sample set

Lab Control Standard: A non site sample created in the laboratory to which a known amount of target analyte is added for assessment of analyte recovery efficiency

Lab Control Standard Duplicate: An exact copy of the Lab Control Standard to further assess analyte recovery efficiency

Matrix Spike: A site sample to which a known amount of target analyte is added for assessment of analyte recovery efficiency.

Matrix Spike Duplicate: An exact copy of the Matrix Spike utilizing the same site sample and known amount of target analyte for assessment of analyte recovery efficiency

ASE (Accelerated Solvent Extractor): A machine that utilizes solvents at high temperature and pressure to extract soil/sediment/ solid samples for PCB analysis

Surrogate Compound Spike: In chemical composition and chromatography similar to the analytes of interest. Usually not found in environmental samples. These compounds are spiked into all samples, standards, blanks, and matrix spike samples prior to analysis. Percent recoveries are calculated for each surrogate.

QC-Quality Control: A set of measures for each sample within an analysis methodology

to assure that the process is in control.

7.0 INTERFERENCES Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of certain plastics. Phthalate esters respond on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Samples and extracts should not be exposed to plastics such as gloves, tubing, coating on clamps, and pipette bulbs, etc.

8.0 SAFETY The chemist should have received in-house safety training and should know the location of first aid equipment and the emergency spill/clean-up equipment, before handling any apparatus or equipment. Safety glasses and protective exam gloves must be worn when handling glassware and samples. Polychlorinated biphenyls have been tentatively classified as known or suspected carcinogens. The chemist must review the Material Safety Data Sheets (MSDS) for PCBs and all reagents used in the procedure before handling them. All solvents should be handled within a lab fume hood.

9.0 EQUIPMENT AND SUPPLIES

9.1 Cell Body: ASE 200 (Accelerated Solvent Extractor) Dionex, 22ML #048821, 33ML #048822.

9.2 Cell caps: Dionex #049450.

9.3 Steel Rod: Used to compresses sample in the cell.

9.4 Metal spatula.

9.5 Mixing Tray: Used to mix sample prior to weighing sample.

9.6 Analytical Balance: Mettler PL-303 used to determine sample mass.

9.7 Cellulose Filter: Prevents the frits of the cell end pieces from being clogged during ASE extraction.

9.8 Turbo Vap Evaporator: Zymark #ZW640-3.

9.9 Turbo Vap Evaporator concentrator tubes: Zymark 250ML, 0.5ML endpoint.

9.10 Turbo Vap LV.

9.11 60 ml ASE vials.

9.12 Vials: glass, 40mL & 4 dram (with Polyseal sealed cap) (20 ML & 10 ML) capacity, for sample extracts.

9.13 Vial Rack: Plastic rack used to hold vials, during all phases of the extract processing.

9.14 Centrifuge: International Equipment Co., Model CL. (or equivalent)

9.15 Wrist Shaker: Burrell wrist action shaker, Model 75 and 88. (or equivalent)

9.16 Pipettes: S/P Disposable Serological Borosilicate Pipettes.
1. 1ML X 1/10 #P4650-11X
2. 5ML X 1/10 #P4650-15
3. 10ML X 1/10 #P4650-110
Kimble Pasteur Borosilicate glass pipette 9" #72050 (or equivalent)

9.17 Beakers: Assorted Pyrex: 250ML, 600mL, and 1000mL.

10.0 REAGENTS AND STANDARDS

10.1 Hydromatrix: Varian #0019-8004.

10.2 Sodium Sulfate: Anhydrous (12-60 Mesh) Used for the laboratory method blank and laboratory control spike.

10.3 Hexane: High Purity Solvent Baxter (Burdick/Jackson) #UN1208. (or equivalent)

10.4 Acetone: High Purity Solvent Baxter (Burdick/Jackson) #UN1090. (or equivalent)

10.5 1:1 Hexane/Acetone: 50%/50% by volume solvent mixture prepared in the lab.

10.6 Florisil: deactivated, solvent washed with 1:1 hexane/ether, baked at 130° C for 16 hours. Deactivated with D.I. water. EM Science #FX0282-1.

10.7 TBA Reagent: Tetrabulylammonium Hydrogen-Sulfite Reagent. (prepared in the laboratory)

10.8 Mercury: Triple distilled Mercury Refining Co, Albany, NY #328502. (or equivalent)

10.9 Sulfuric Acid: Na_2SO_4 (concentrated) Mallinkrodt #2468 #UN1830. (or equivalent)

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT and STORAGE

11.1 All soil samples should be collected in 4 oz or 8 oz jars with Teflon lined lids.

11.2 All soil samples should be stored in the walk in refrigerator at 4 + or – 2 degrees C before extraction.

11.2 Soil samples have a hold time of 14 days before they need to be extracted.

11.3 The extracted solvents must be analyzed within 40 days of their extraction date.

12.0 QUALITY CONTROL

Every sample set is run with two control samples: the blank standard and the lab control standard

12.1 The extraction chemist should have completed an acceptable demonstration of precision and accuracy before performing the method without supervision. The addition of spiking material to a sample or blank must be witnessed by another extraction chemist and signed in the extraction logbook. All surrogates and matrix spikes must meet acceptable QC limits.

12.2 A method blank sample and lab control spike must be prepared per each extraction batch or 1 per 20 site samples whichever is more frequent. A matrix spike/matrix spike duplicate (or lab duplicate) should be prepared for every 20 site samples or as per client specified quality assurance project plan (QAPP). Spike default for LCS, MS, MSD is 1.0mL of A1242 @ 0.5ppm. Client and/or project specifications may dictate alternate amount or Aroclor.

12.3 PCB Surrogates TCMX and DCBP are added to each sample prior to extraction to measure extraction/cleanup efficiency. Default surrogate is: 1.0mL of 0.05ppm TCMX / 0.5ppm DCBP. Client and/or project specifications may dictate alternate amount.

13.0 CALIBRATION AND STANDARDIZATION

Please see determinative method (Lab SOP NE148) for details

14.0 PROCEDURES

14.1 Sample Preparation

14.1.1 Throughout the entire process it should be noted that if the chemist encounters any problems or difficulties with any samples or steps involved, all work should STOP! Any problems should be brought to the attention of the supervisor and documented in the extraction logbook.

14.1.2 Before any steps are taken, the chemist should first review the sample job folder and fill in the appropriate spaces on the internal sample tracking form and initial.

14.1.3 If the sample is sediment and contains a water layer, decant and discard the layer as aqueous PCB waste. Mix sample thoroughly in the sample container. If there is not enough room to mix sample in the container then remove sample from container, using a metal spatula, into a mixing tray. Mix the sample thoroughly and discard any foreign objects such as sticks, rocks or leaves. Note however that the sample may be composed entirely of rock, concrete or some other solid material in which case the entire sample is treated as the solid.

14.2 Sample Extraction

14.2.1 Setup one 4 oz. jar for each sample. Pick the first sample, label a beaker with the sample number, and tare the beaker. Using a metal spatula, add 10 g to 11 g of the wet sample to the beaker. Use the sodium sulfate as the sample for the Blank and Laboratory Control Sample. Record the weight in the sample mass book.
NOTE: ALL SAMPLE CONTAINERS ARE TO BE RETURNED TO THE APPROPRIATE REFRIGERATOR. FOR ALL EMPTY SAMPLE CONTAINERS, SEE THE CHEMICAL HYGIENE PLAN FOR PROPER DISPOSAL.

14.2.2 If the PCB concentration is to be determined on a dry weight basis the percent total solid must be determined. Weigh approximately 5 grams of the previously homogenized sample in a previously weighed, tarred aluminum-weighing pan. Record the weight of the sample and the tare weight of the pan in the percent total solids log. Place the sample in a drying oven at 100 to 110 degrees Celsius for at least 4 hours. Record the time placed in the oven and the oven temperature in the LIMS system. Remove the samples from the drying oven and allow to cool. Weigh the pan and sample (see calculations on how to calculate percent solids).

14.2.3 Before the sample is added to the cell, the sample must be dried. The sample is dried by adding Hydromatrix. The amount of this drying agent being used depends on how much water is in the sample. The more water present in the sample, the more drying agent will be needed to dry the sample. Mix the sample and drying agent thoroughly with a metal spatula.

14.2.4 The cell size to be used during the extraction will be determined by the final volume of the sample after the drying agent has been added. **NOTE: sometimes the sample will have to be separated into two cells for the extraction if too much drying agent has been added.**

14.2.5 Select the appropriate cell body size for each sample. Assemble one cell end cap to the cell body. Place 2 cellulose filters into the open end of the cell and push it down to the cell end cap using the black ASE push rod.

14.2.6 Label cells with the sample number. Label the corresponding 60 voa vials on the base of the vial.

14.2.7 Place the cell into a clean mixing pan. Add the dried extract to the cell using the metal spatula to guide the sample into the cell. Any sample that fell outside of the cell will be collected in the mixing tray. Remove the cell from the mixing tray and added the sample that is in the mixing tray to the cell. You may need to compact the sample in the cell, using the steel rod, while the sample is being added. **NOTE: rinse the steel rod with acetone and Dichloromethane before using on a different sample or placing it in the storage drawer.**

14.2.8 Add surrogate and matrix spiking compounds at this point.

14.2.9 Assemble the top cell end cap to the cell, hand tighten. Place the first cell to be extracted in the 1 position on the cell tray (top tray) and the 60mL vial in the 1 position on the vial tray (bottom tray). The cells and 60mL vials for each sample must be in the same number position on the two trays.

14.2.10 Solvent used for PCB extraction is 1:1 Hexane/Acetone

14.2.11 Select the appropriate method or schedule and start the ASE.

14.2.12 When the extraction program is complete, transfer the hexane layer (top layer) using a 10mL pipette into a prerinsed turbo tube. Leaving only the water layer in the 60mL vial. Leave the 10ML pipette in the turbo tube.

14.2.13 Rinse the 60mL vial using 5 pipettes of hexane. Hand shake for 30 seconds. Allow the two layers to separate, and pipette the hexane layer, using the same 10ML pipette, into the turbo tube. Repeat this step 1 more time for a total of 2 hexane extractions on the water layer.

14.2.14 Rinse the 10ML pipette with two pipettes of hexane on the outside of the 10ML pipette that was in contact with the sample extract and two pipettes of hexane through the 10ML pipette and collect into the turbo tube.

14.2.15 All glassware must be rinsed with technical grade (tech)-acetone or a "for rinsing-only" labeled solvent, and dried in the hood before other cleaning steps.

14.3 Solvent Reduction: TurboVap Evaporator System

14.3.1 The Turbovap evaporator system is used in place of the Kuderna Danish (KD)-concentrator

apparatus. The turbovap uses a heated water bath and positive pressure nitrogen flow/vortex action. The unit maintains a slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract which allows fractional reduction of the solvents without loss of higher boiling point analytes.

14.3.2 Turn the unit on (switch is located on the backside of the unit) and allow to heat up to the specified temperature for individual solvent use.
This is indicated by the "Heating" display light, located above the temperate control knob on the right side of the unit. The system is at the proper temperature when the "At Temperature" light is lit. This is located above the "Heating" display light.

14.3.3 As a precaution the TurboVap system regulators should be checked to assure that no residual gas pressure remains within the system and that the gas cylinder valve and gas pressure regulators are both off before placing samples in the apparatus. Residual gas pressure may cause splashing and cross contamination of samples. To bleed the system of residual gas pressure place an empty turbo tube into the water bath and close the lid. Make sure that the nitrogen gas cylinder valve is turned off and slowly turn on the gas pressure regulator. Bleed any residual gas until the regulator output pressure gauge reads "0" psi. Proceed to 8.3.4 . Make sure to wipe down all surfaces with hexane before concentration samples.

14.3.4 Place the turbo tube containing the samples into the TurboVap and close the lid. Turn on the gas cylinder valve first and then begin slowly turning the pressure regulator on. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the reduction.

14.3.5 Turbo Vap – LV low volume unit:
Turn the unit on (switch is located on the left side of the unit) and allow it to heat up to 40 degrees Celsius. This is indicated by the "Concentrating with Turbo Vap LV" blinking number under the temperature control button. When the system is at the proper temperature the number will no longer blink. As a precaution the Turbo Vap LV regulator should be checked to assure no residual gas remains in the system. Residual gas may cause splashing and cross contamination of the samples. To resolve this place a vial into the vial in to the turbo vap and close the lid. Press the start button and proceed to turn the gas regulator knob counter-clockwise until the regular reads zero. Place the 60 ml vials into the turbo vap. Press the button to turn on the appropriate row of stations that are being used. The press the start button and adjust the regulator until the samples begin to swirl. Check the sample every few minutes and adjust the gas to keep the samples swirling.

14.3.6 The process for solvent (hexane/acetone) reduction takes approximately 20-30 minutes. Do not leave the unit unattended as extracts may be blown to dryness and PCB loss may occur. Immediately notify a supervisor if an extract is blown to dryness. Concentrate the solvent to approximately 10.0mLs and fill back up with hexane and restart the solvent reduction (this step is done to make sure there is no acetone left over in the samples).

14.3.7 Concentrate the solvent to approximately 10.0mL. Remove the samples from the TurboVap and place in the rack. The remaining solvent will consist largely of hexane

since the acetone component is fractionally removed at a faster rate than hexane. NOTE: Not all samples will evaporate at the same rate; sample extracts containing large amounts of petroleum or other non-volatile liquids may stop reducing before the 10.0mL point is achieved. Samples that stop reducing should be removed as soon as possible.

14.3.8 Quantitatively transfer the sample extract with a pasteur pipette into an appropriate volumetric flask (25mL for soil extracts). Rinse the turbo tube or vial with 3 pasteur pipettes of hexane, then transfer the hexane rinse to the volumetric. Repeat the hexane rinse two more times for a total of three hexane rinses of the turbo tube. After the sample has been transferred, rinse the pasteur pipette with 0.5mL of hexane into the volumetric flask. Add hexane to the volumetric meniscus mark.
Invert the volumetric flask at least three times to mix completely. Decant the contents into a pre-labeled 40mL vial.

14.3.9 All dirty glassware must be rinsed with tech-acetone or a "For Rinsing-Only" labeled solvent and dried in the fume hood before being washed.

14.4 Sample Extract Cleanup

14.4.1 Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection contain co-extracted xenobiotics and other interfering substances which must be removed before accurate chromatographic analysis can be performed.

14.4.2 Not all clean-up procedures need to be performed on every sample and several are sample matrix specific. The experience of the analyst combined with the sampling site history should guide the selection of which clean-up procedures are necessary. The sequence and number of replicates of cleanup steps performed are recorded by the sample preparation chemist on the sample tracking log sheet. Sample extract cleanups are performed on set volume extracts. The set volume is 25 ML for soil/sediment/solid samples.

14.4.3 Sulfuric Acid Wash

14.4.3.1 The concentrated sulfuric acid treatment removes hydrocarbons and other organic compounds, which are co-extracted with the PCB residues.

14.4.3.2 Add 5.0mL concentrated H_2S0_4 and shake for 30 seconds by hand, centrifuge for approximately 1 minute, transfer approximately 20MLs of the hexane upper layer to a 40mL vial.

14.4.3.3 Repeat 8.4.3.2 if the sample extract appears to be heavily loaded (opaque) with colored material. Two to three acid washes may be required. Note: it is entirely possible that all colored material will not be removed from the extract.

14.4.4 Elemental Sulfur Clean-up

14.4.4.1 Elemental sulfur is soluble in the extract solvents used for sediment and soil samples. It is commonly found in many sediment/soil samples, decaying organic material, and some industrial wastes.
Large amounts of sulfur can cause the electron capture detector (ECD) to signal saturate for long periods during the elution envelope of PCBs. Even small amounts of sulfur can interfere with PCB measurement as a co-eluting chromatographic peak.

14.4.4.2 Two techniques exist for the elimination of elemental sulfur in PCB extracts. Mercuric precipitation (Mercury Shake) and the Tetrabutylammonium (TBA) sulfite procedure.

Tetrabutylammonium sulfite causes the least amount of degradation to a broad range of pesticides and organics compounds, while mercury may degrade organophosphorus and some organochlorine pesticides. The TBA procedure also has a higher capacity for samples containing high concentrations of elemental sulfur.

14.4.5 Removal of Sulfur Using Mercury

14.4.5.1 Mercury is a highly toxic metal. All operations involving mercury should be performed within a hood. Prior to using mercury, the chemist should become acquainted with proper handling and emergency spill/clean-up procedures associated with this metal and must have reviewed the material safety data sheet for mercury.

14.4.5.2 Add 1-3 drops of mercury to the sample extracts, cap, and place on the wrist shaker for 30 minutes. The sulfur is converted to mercuric sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental sulfur.

14.4.5.3 Remove the sample extracts from the wrist shaker and place in the centrifuge for 2 minutes on speed setting on #4.

14.4.5.4 Transfer the sample extract to a clean 40mL vial.

14.4.5.5 The precipitated sulfur can be removed from the extract by performing a sulfuric acid clean-up or a Florisil slurry.

14.4.6 Removal of Sulfur using TBA Sulfite

14.4.6.1 The TBA procedure removes elemental sulfur by conversion to the thiosulfate ion, which is water-soluble.

14.4.6.2 Add 2.0mL TBA Sulfite Reagent, 1.0mL 2-propanol, and approximately 1 scoop (using the dedicated dispenser) of sodium sulfite crystals to the extract and shake for at least 5 minutes on the wrist shaker and observe. An excess of sodium sulfite must remain in the sample extract during the procedure. If the sodium sulfite crystals are entirely consumed add one or two more aliquots to the extract and observe.

14.4.6.3 Place the samples on the wrist shaker for 45 minutes observing at 15-minute intervals to make sure that the sodium sulfite is not consumed. Add 5mL organic free water and shake for 15 minutes.

14.4.6.4 Place the samples into the centrifuge and spin for approximately 2 minutes on setting #4.

14.4.6.5 Transfer the hexane layer to a new 40mL vial and cap.

14.4.7 Florisil Adsorption (Slurry)

14.4.7.1 The Florisil slurry removes co-extracted polar compounds, residual water, and residual acid and is recommended as the final cleanup step before the extract is submitted for GC analysis.

14.4.7.2 Add approximately 1.5 grams of tested and approved deactivated Florisil to each vial containing the sample extract. **SEE SUPERVISOR FOR THE APPROPRIATE FLORISIL DEACTIVATION CONCENTRATION TO BE USED.**

14.4.7.3 Vigorously shake the vial for approximately 1 min by hand or on the wrist shaker.

14.4.7.4 Place the vial(s) into the centrifuge for 2 minutes on setting #4.

14.4.7.5 Transfer the extract to a clean 40mL vial.

14.5 Extract Screening and Dilution:

14.5.1 PCB extracts are generally screened by GC to determine the approximate concentration before final analysis. Prior site history and client supplied estimates of sample concentration may be used to determine what, if any, extract dilution is necessary. Extracts of unknown concentration are generally screened at a 10 to 100-fold dilution.

14.5.2 The supervising chemist is responsible for determining initial screening dilutions. Extract dilutions are prepared by transferring an aliquot of the original sample extract into a vial containing the correct amount of "make up" volume of hexane. For example, a 1 to 10 dilution is performed by adding 1.0mL of the extract to 9.0mL hexane. The vial containing the diluted extract is labeled denoting the equivalent extract volume after the dilution; e.g. a 25ML extract diluted 1 to 10 is labeled "250X", an undiluted 25ML extract is labeled "25X". When high dilutions are prepared, secondary (serial) dilutions of the initial diluent are prepared; e.g. a 100-fold dilution is prepared by a 1 to 10 dilution of the initial extract, then a 1 to 10 dilution of the resulting diluent.

14.5.3 Perform the dilution using an appropriate class A disposable volumetric pipette to transfer the extract and for the make-up volume of hexane. Make sure that the vial is properly labeled. Cap and invert the vial at least three times to thoroughly mix the extract with the solvent.

14.5.4 Transfer 1mL of the extract to a labeled 1.5mL GC autosampler vial. Record the sample data on the LIMS GC Queue Lab Sheet. Submit the GC Queue Lab Sheet with the sample extracts to the GC analyst.

15.0 CALCULATIONS

Calculate the percent solids by:

$$\frac{\{(\text{wt. of pan + dried sample}) - (\text{wt.of pan})\} \times 100}{(\text{wt. of wet sample})}$$

16.0 METHOD PERFORMANCE

Please see determinative method (Lab SOP NE148) for details.

17.0 POLLUTION PREVENTION

Please see determinative method (Lab SOP NE148) for details.

18.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

Please see determinative method (Lab SOP NE148) for details.

19.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

Please see determinative method (Lab SOP NE148) for details.

20.0 CONTINGINCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Please see determinative method (Lab SOP NE148) for details.

21.0 WASTE MANAGEMENT

see NEA054.SOP, NEA083.SOP, NEA089.SOP

22.0 REFERENCES

1. U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

2. "Guide to Environmental Analytical Methods", Third Edition, Genium Publishing Corporation, 1996 .

23.0 TABLES, DIAGRAMS, FLOWCHARTS and VALIDATION DATA

1. ASE Methods
2. ASE control panel keypad.
3. ASE cell cleanup procedure.

ASE METHODS

METHOD #	ANALYTE OF INTEREST	MATRIX	REQUIRED SOLVENT *	AMOUNT OF SAMPLE
1	PCB	WIPE	1	WIPE
2	PET I.D.	SOIL	1	10 g
2	8270	SOIL	3	30 g
3	**PCB**	**SOIL**	**2**	**10 g**
4	22mL Cell cleaning	NONE	3	NONE
5	33mL Cell cleaning	NONE	3	NONE
6	8081 Pest	Soil	2	10g

*REQUIRED SOLVENT CHART

1 - HEXANE
2 - 1:1 HEXANE / ACETONE
3 - 1:1 DICHLOROMETHANE / ACETONE

CONTROL PANEL KEYPAD

* TRAY	Tray is in free spin for manual turning.
TRAY *	Tray drive mechanisms are engaged and cannot be moved manually.
RINSE	Starts a manual rinse cycle.
* START	System is idle.
START *	system is currently running a method or schedule.
ABORT	Interrupts current run. Continue with abort function to terminate ASE run.
MENU	Displays a list of available screens.

* - LIGHT IS ON

ASE cell cleanup procedure

Remove the end caps of ASE cells. Using a metal spatula designated for cell clean up, push the extracted sample out of the cell into a garbage can. Wash the interior and exterior of the cell and cell end caps with soap and water (use the brush designated for ASE use only). Dry the cell parts with a paper towel and reassemble the cell.
Run the washed cells on the ASE (use a new 60mL vial for each cell) using method 4 for 22ml cells, 5 for 33 ml cells and 1:1 dichloromethane/acetone as the solvent.

NOTE: After the cells has been used 20 times or if the frits become clogged, the cell end caps should be taken apart and sonicated for 10 minutes in acetone and 10 minutes in dichloromethane.

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STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF POLYCHLORINATED BIPHENYL (PCB) AROCLORS BY US-EPA SW-846 METHOD 8082

SOP Name: NE148_06.SOP
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STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF POLYCHLORINATED BIPHENYL (PCB) AROCLORS BY US-EPA SW-846 METHOD 8082

1.0 Identification of Test Method

1.1 This method is used to determine Polychlorinated Biphenyl (PCB) by gas chromatography with electron capture detection and Total Aroclor Quantification using EPA SW 846 Method 8082- Polychlorinated Biphenyl (PCB) Aroclors by Capillary Column GC.

2.0 Applicable Matrix

2.1 This SOP is applicable in the determination and quantification of PCBs as outlined in EPA SW-846 Method 8082. It is applicable to the following matrices: water, soil, sediment, sludge, oil, fuel oil, waste solvent, fish, other aquatic animals, tissue samples, and air cassette samples including polyurethane foam (PUF) and associated filters for EPA Methods TO-4A and TO-10A.

3.0 Detection Limit

3.1 Detection Limit: Reporting Limits (RLs) and Method Detection Limits (MDLs) vary for each compound.

3.1.1 The following are default Reporting Limits based on the lowest calibration standard and global MDL /Reporting Limits based on MDL studies performed by NEA used for EPA Method 8082. See attachment G for example of MDL study. Reporting Limits (PQLs) are based on the Lowest Calibration Standard.

Matrix	Sample Mass/Volume Extracted	Calibration Curve Low Standard	Extract Volume	Global MDL* (All Aroclors)	RL (PQL) (all Aroclors)
Soil/Sediment/Solid	10 g	20 ng/ml	25 mL	0.0115 mg/kg	0.050 mg/kg
Water	1 Liter	5 ng/ml	10 mL	0.0075 ug/L	0.050 ug/L
Biota	10 g (wet weight basis)	20 ng/mL	25 mL	0.0104 mg/kg	0.050 mg/kg
Polyurethane Foam Cassette (TO-4A/TO 10A)	1 PUF	20 ng/ml	5 mL	0.021 ug/Puf	0.100 ug/Puf
Waste Oil	0.5 g	20 ng/ml	25 mL	0.200 mg/kg	1.00 mg/kg
Wipe	1 Wipe	20 ng/ml	25 mL	0.115 ug/Wipe	0.500 ug/wipe

3.2 Individual MDLs and RLs are determined every two years for each instrument with matrix specific MDL studies for each extraction methodology. MDLs must be determined again whenever a major change in instrumentation or extraction methodology occurs.

3.3 MDLs are verified annually by the extraction and analysis of a low level MDL verification check sample. The Aroclor must be observed qualitatively in the MDL verification check sample.

4.0 Scope and application, including components to be analyzed

4.1 This SOP is applicable in the determination and quantification of PCB as outlined in EPA SW-846 Method 8082. It is applicable to the following matrices: water, soil, sediment, sludge, oil, fuel oil, waste solvent, fish, other aquatic animals, tissue

samples, wipes and air cassette samples including polyurethane foam (PUF) and associated filters for EPA Methods TO-4A and TO-10A.

4.2 In general, samples are extracted, or in the case of oils and waste solvent diluted, with a pesticide grade solvent. Applicable extraction methods for solids and animal tissues include: SW-846 Method 3540 (Soxhlet), SW-846 Method 3545 (Pressurized Fluid Extraction), or SW-846 Method 3550 (Ultrasonic Extraction). Extraction methods for aqueous samples include SW-846 Method 3510 (Separatory Funnel), and SW-846 Method 3520 (Continuous Liquid Liquid Extraction). The extracts are further processed by concentrating or diluting, depending on the PCB concentration, and carried through a series of clean-up techniques. Applicable cleanup techniques include SW-846 Method 3620 (Florisil Cleanup), SW-846 Method 3660 (Sulfur Cleanup) and SW-846 Method 3665 (Sulfuric Acid/Permanganate Cleanup). The sample is then analyzed by direct injection onto a gas chromatographic system and detected by an electron capture detector.

4.3 This method provides detailed instructions for gas chromatographic conditions, calibration, and analysis of PCB by capillary column gas chromatography. Each matrix requires different sample handling or special preparation procedure before analysis can be performed. Each sample matrix will be covered separately in the extraction standard operating procedures.

5.0 Summary of Test Method

5.1 Samples are extracted with a pesticide analytical grade solvent. The extracts are further processed by concentration and a series of clean-up procedures. The sample extracts are then analyzed by injecting onto a gas chromatographic system and with an electron capture detector in series.

5.2 The purpose of this SOP is to provide a detailed written document for quantification of PCB according to SW-846 Method 8082 specification.

5.3 This SOP provides detailed instructions for gas chromatographic conditions, calibration, and analysis of PCBs by gas chromatography. Sample extraction and cleanup procedures are described separately in additional laboratory Standard Operating Procedures.

5.4 The following PCB Aroclors can be determined by this method*:

Compound	CAS Number
Aroclor 1016	12674-11-2
Aroclor-1221	11104-28-2
Aroclor-1232	11141-16-5
Aroclor-1242	53469-21-9
Aroclor-1248	12672-29-6
Aroclor-1254	11097-69-1
Aroclor-1260	11096-82-5

*Note: Additional PCB Aroclor mixtures including Aroclor 1262 and Aroclor 1268 may be analyzed by this method with minor modifications

5.4 Extensive knowledge of this SOP and EPA Method 8082 is required. The analysis portion of this method should be performed by a skilled chemist or by an analyst trained in the quantification of trace organics by gas chromatography.

6.0 Definitions

6.1 Accuracy – The degree of conformity of a measured or calculated quantity to its actual (true) value. Accuracy is assessed by analysis of references samples and percent recoveries.

6.2 Analytical Batch –The basic unit for analytical quality control is the analytical batch, which is defined as samples which are analyzed together with the same sample method sequence, lots of reagents, and manipulations of samples can occur within the same time period or in continuous sequential time periods. Samples in each batch should be of similar matrices (e.g. water, sediment, soil, etc.).

6.3 Blank – A blank is an artificial sample designed to monitor the introduction of artifacts into the process. For aqueous samples, reagent water is used as a blank matrix, however, a universal blank matrix does not exist for solid samples, but sometimes sodium sulfate is used as a blank matrix. The blank is follows the all the steps of the method. A reagent blank is an aliquot of analyte-free water or solvent analyzed with the analytical batch. Field blanks are aliquots of analyte-free water or solvents brought to the field, and are sealed containers which are transported back to the laboratory with the sample containers. Trip blanks and equipment blanks are two specific types of field blanks. Trip blanks are not opened in the field. They are a check on sample contamination originating from sample transport, shipping and from site conditions. Equipment blanks are opened in the field, and the contents are poured appropriately over or through the sample collection device, which is collected in a sample container, and returned to the laboratory as a sample. Equipment blanks are a check on sampling device for cleanliness.

6.4 Continuing Calibration Check Standard (CCCS) –The continuing calibration check standard contains all target analytes found in the calibration standards and is used to verify that the initial calibration is prepared correctly, and that the instrument system is correctly calibrated. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards.

6.5 Calibration Standard (ICAL)– A series of known standard solutions used by the analyst for instrument calibration. Calibration standards are prepared from a primary standard and/or stock standard solutions.

6.6 CAS Number – An assigned number used to identify a chemical. CAS stands for Chemical Abstracts Service, an organization that indexes information published in Chemical Abstracts by the American Chemical Society and provides index guides by which information about particular substances may be located in the abstracts. Sequentially assigned CAS numbers identify specific chemicals, except when followed by an asterisk (*), which signifies a compound (often naturally occurring) of variable composition. The CAS numbers have no chemical significance, rather it is a concise, unique means of material identification. (Chemical Abstracts Service, Division of American Chemical Society, Box 3012, Columbus, OH 43210: [614] 447-3600).

6.7 Duplicate– A second aliquot of a sample that is treated in the same manner as the original sample in order to determine the precision of the method.

6.10 Environmental Sample – An environmental sample or field sample is a representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which the determination of a composition or contamination is requested or required.

6.11 Initial Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the analytical detector or method.

6.12 Instrument Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

6.13 Laboratory Control Sample (LCS) – Also known as the Quality Control (QC) Check Standard or Quality Control (QC) Check Sample. The LCS consists of an aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added. The LCS is extracted and analyzed exactly like a field sample, and its purpose is to determine whether the analysis is in control, and whether the laboratory is capable of making accurate and precise measurements.

6.14 Laboratory Method Blank – An analytical control consisting of all reagents and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

6.15 Matrix – The predominant material of which the sample to be analyzed is composed. Matrix is not synonymous with phase (liquid or solid).

6.16 Matrix Spike – Aliquot of sample (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

6.17 Matrix Spike Duplicate – A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

6.18 Method Detection Limit (MDL) – The minimum constituent concentration that can be measured and reported with 99% confidence that the signal produced is different from the blank in a given matrix. The MDL is determined from a minimum of seven replicate samples, taken through the entire preparation and analysis procedure. The standard deviation (*s*) of those replicates is multiplied by a student's t-factor in order to calculate the MDL.

6.19 MSDS – Material Safety Data Sheet. OSHA has established guidelines for the descriptive data that should be concisely provided on a data sheet to serve as the basis for written hazard communication programs.

6.20 PCB Polychlorinated biphenyls (PCBs) are a class of 209 individual chemical compounds (congeners), in which one to ten chlorine atoms are attached to a biphenyl molecule. The use of PCBs such as in manufacturing, has made them a frequent environmental pollutant.

6.21 Precision – The agreement between a set of replicate measurements without assumption of knowledge of the true value. Precision is assessed by means of a duplicate or replicate sample analysis.

6.22 Quality Control – Set of measures within a sample analysis methodology to assure that the process is in control.

6.23 Standard Curve – A curve which plots concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by diluting the stock analyte solution in graduated amounts which cover the expected range of the samples being analyzed. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards must be prepared using the same type of acid, solvent, and concentration, as well as the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

6.24 Stock Solution – Standard solutions which can be diluted to derive the other standards.

6.25 Surrogate – Organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, calibration and check standards, samples (including duplicates and QC reference sample) and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

6.26 Surrogate Standard – A pure compound added to a sample in the laboratory just before processing so that the overall efficiency of a method can be determined.

7.0 Interferences

7.1 Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing. Samples and extracts should not be exposed to plastic materials. Phthalate esters exhibit a response on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Laboratory method blanks must be thoroughly reviewed for the presence of non-target peaks and samples must be compared with blank chromatographic patterns.

7.2 Elemental sulfur (S_8) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur can be removed through the use of clean-up procedures, Method 3660.

7.3 Polychlorinated Terphenyls (PCTs), Polybrominated Biphenyls (PBB), Polychlorinated Naphthalenes (PCN), as well as Polychlorinated Dibenzo-p-Dioxins (PCDDs) can co-elute with PCBs. Interference from these compounds, when in high concentration, is common if clean-up procedures are not followed. These materials may be removed through the use of specified clean-up procedures when presence is suspected.

7.4 Organochlorine pesticides can be a source of interference when co-extracted with samples submitted for PCB analysis. Caution must be used when processing chromatographic data so that pesticides do not interfere with PCB quantification peaks. Specified clean-up can be followed to eliminate many pesticides when analyzing PCBs.

8.0 Safety

8.1 Safety glasses and disposable gloves must be worn when handling samples and extracts.

8.2 All manipulations of sample extracts should be conducted inside a chemical fume hood. Manipulation of sample extracts outside of a fume hood should be minimized by the analyst.

8.3 Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets to familiarize themselves with the precautions for handling solvents and chemicals used to process samples. The analyst should refer to the laboratory chemical hygiene plan for further safety information.

8.4 Samples remaining after analysis should either be returned to the customer for disposal or disposed of through the laboratory's disposal plan. Refer to the sample custodian for assistance and also standard operating procedure NE054, for disposal of laboratory waste.

9.0 Equipment and Supplies

9.1 Gas Chromatograph: Complete system for high resolution, capillary column capability and all required accessories. Northeast Analytical, Inc. will use a Varian Model 3400 or 3800 (or equivalent) gas chromatograph (or equivalent), equipped with a Model 1077 or 1177 split/splitless injector (or equivalent), temperature programmable oven, Varian Model 8200, Varian Model 8400, or LEAP GC pal automatic sampler (or equivalent), and electron capture detector (or equivalent). A data system and integration of detector signal is interfaced to the gas chromatograph.

9.2 Chromatographic Data System: A data system for measuring peak height and peak area. The Empower computer network based workstation (Waters Corporation) is employed to capture detector response and digitally stores the chromatographic, electronic peak integration for precise calculations, database structuring of the analytical information, and archival capabilities.

9.3 NEA LIMS: This is a custom in house Laboratory Information Management System (LIMS) developed by Northeast Analytical, Inc. with the intent of handling all aspects of the laboratory environment. All relevant sample data such as initial information from client, extractions, analysis, and final reporting are captured electronically and stored in a relational database. From this database, a variety of reports can be generated based upon the packaging requirements requested.

9.4 Column (Primary): ZB-1, Phenomenex Cat. No. 7HG-G001-11; 30 m x 0.25mm x 0.25 um ; DB-1, J&W Part No. 122-1032; 30 m x 0.25 mm x 0.25 um ; or equivalent.

9.5 Column (Secondary): ZB-5, Phenomenex Cat. No. ZB-5-G002-11; 30m x 0.25mm x 0.25 um ; DB-5, J&W Part No. 122-5032; 30m x 0.25 mm x0.25 um; or equivalent.

9.6 Class A volumetric flasks: 5.0 – 100mL.

9.7 30ml vials and 15 ml vials for sample extract storage.

9.8 Pasteur pipettes.

9.9 250ml and 100ml beakers, glass.

9.10 Disposable 1.0, 5.0, and 10.0 ml pipettes.

9.11 Hexane, Burdick and Jackson-Pest Grade.

9.12 Acetone, Burdick and Jackson.-Pest Grade.

9.13 Toluene, Baker, (Cat.No. 9336-03).

9.14 Methylene Chloride, Burdick and Jackson, (Cat. No. 300-4).

9.13 Ferrules: 0.4 mm graphite/vespel, Restek 20229, and ¼" graphite ferrules, Restek 20210, or equivalent.

9.14 Injector septa: Thermolite Septa, Restek 20365, or equivalent.

9.15 Injector liner: Low Pressure Drop Liner w/Glass Wool, Restek 21033, or equivalent.

9.16 SGE Injector Syringe 10.0 µL: SGE 002987, or equivalent

9.17 Auto sampler vials: Snap vial 12x32mm Clear w/P, Microliter 11-5200

9.18 Snap Caps: 11mm Natural Snap Cap PTFE, Microliter 11-0051N-B.

10.0 Reagents and Standards

10.1 Analytical Standard Solutions.

10.1.1 Aroclor Stock Standard Solutions

10.1.1.1 Polychlorinated Biphenyls - Neat commercial material for standard preparation. These materials are multi-component mixtures of PCB congeners and are the actual materials that were used in products such as electric power transformers and capacitors. Monsanto was the largest producer of PCB formulations and sold them under the trade name Aroclor. These standards should be compared to PCB reference materials to verify commercial materials. Aroclors may be used as calibration standards, but they must have the same pattern and congener distribution.

10.1.1.2 Stock standards are prepared from individual Aroclor formulations by weighing an exact amount of the neat material to the nearest 0.1 g, and dissolving and diluting to specified volume in a 100 mL volumetric flask with hexane. See Attachment A, Table 1 for exact weights of each compound.

10.1.1.3 The stock standards are transferred into Boston bottles and stored in a refrigerator at 0-6°C, protected from light.

10.1.1.4 The stock standards are transferred into screw-cap boston bottles and stored in a freezer less than 0°C, but not exceeding 0°C, and protected from light. Stock standards should be checked frequently for signs of evaporation, especially just prior to preparing calibration standards. Stock PCB standards must be replaced after one year, or sooner if a problem with instrument calibration is detected.

10.2 Calibration Standards

10.2.1 Calibration standards are prepared at five concentration levels using a prepared working standard. See Attachment A, Tables 2, 3A, AND 3B for the preparation and exact concentrations of the working standards. The following five standards make up the initial calibration curve standard set for a High Level curve : 20 ng/mL, 100 ng/mL, 250 ng/mL, 500 ng/mL, 1000 ng/mL. The following five standards make up the initial calibration curve set for a Low Level curve: 5 ng/mL, 10 ng/mL, 20 ng/mL, 50 ng/mL, 100 ng/mL.

10.2.1 The two surrogates Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) are included in the A1254 calibration standards. The standard for TCMX/DCBP is prepared by diluting 1 mL of TCMX/DCBP custom standard solution (ULTRA, cat.#CUS-4911, at 500/5000 ng/mL) into a 1000 mL volumetric flask resulting in a solution of TCMX/DCBP at 0.5/5.0 PPM

10.2.2 Refer to Attachment A, Tables 4A and 4B for instructions on preparation of the calibration standards containing A1254 and the surrogates. Refer to Attachment A, Tables 3A and 3B for instructions on preparing the remaining calibration standards.

10.2.3 Transfer all calibration standards to 30 ml vials and store in a refrigerator at 0-6°C, protected from light. Calibration standards must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

10.3 Continuing Calibration Standards:

10.3.1 The surrogate compounds Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) are included in all Continuing Calibration Check Standards at a concentration near the mid-point of the surrogate calibration curve sequence. All continuing calibration standards are prepared independently from calibration standards, by using an alternate source purchased from standard vendors. Refer to Attachment B, Tables 1-3 for instructions on preparation of these standards.

11.0 Sample Collection, Preservation, Shipment and Storage

11.1 Sample Collection and Preservation:

11.1.1 Routine soil, sediment, sludge, solid and concentrated liquid samples should be collected in 8 oz clear glass wide-mouth jars, fitted with a Teflon-lined cap. Aqueous samples should be collected in 1 liter amber glass bottles with a Teflon-lined cap. Project specific protocols may require that containers be pre-cleaned to EPA specification protocol. Protect samples from light.

11.1.2 All samples must be placed on ice (≤0°C) or refrigerated at 0-6°C from the time they are collected until delivery to the lab. Samples that are collected within driving distance of the laboratory and delivered the same day may not have reached temperature acceptance limits. These samples are deemed acceptable if evidence of cooling is present (i.e. they are received with ice in the cooler).

11.2 Sample Shipment:

11.2.1 Sample Shipment is accomplished through a carrier such as Federal Express or United Postal Service for overnight 1-day delivery to the lab. Shipment is normally handled by the field personnel collecting the samples and coordinated with sample receiving department at the lab. Samples can also be picked up by the lab courier service if samples are collected within driving distance to the lab.

11.3 Sample Storage:

11.3.1 The samples must be protected from light and refrigerated at 0-6°C from time of receipt until they are removed from storage for extraction. Remaining sample material will be stored protected from light and refrigerated at 0-6°C. Samples will be disposed of or stored or archived according to project specifications.

11.3.2 Routine soil, sediment, sludge, solid, liquid and concentrated liquid samples are stored in a refrigerator dedicated for this type of sample.

11.4 Sample Extract Storage:

11.4.1 Sample extracts must be protected from light and refrigerated at 0-6°C during the analysis. After analysis is complete, sample extracts will be discarded after 60 days or can be archived in a freezer at less than -20-0°C for longer periods of time depending on the program requirements.

11.4.2 Field samples, sample extracts, and calibration standards must be stored separately.

11.5 Required Hold Times

11.5.1 Extraction of solid samples by appropriate technique must be completed within fourteen days from sample collection.

11.5.2 Extraction of aqueous samples by appropriate technique must be completed within seven days from sample collection.

11.5.3 Sample extracts must be analyzed within forty days of sample extraction.

12.0 Quality Control

12.1 This section outlines the necessary quality control samples that need to be generated at the time of sample extraction. The results of the quality control measurement samples document the quality of the data generated. The following table lists the Quality Control Requirements for capillary gas chromatography analysis of PCBs.

Quality Control Requirements

QC Sample	Frequency
Method Blank	With each sample batch (up to 20 samples).
Lab Control Spike	With each sample batch (up to 20 samples).
Cont Cal Check Std	Analyzed prior to each sample batch and at a Frequency or one per ten injections. Each analytical sequence must close with a Continuing Calibration Check Standard (CCCS).
Duplicate Analysis	Field generated sample – analyzed at discretion of client.
Matrix Spike	One matrix spike per 20 field samples or designated sample batch may be performed as specified in the client site plan.
Matrix Spike Duplicate	One matrix spike duplicate per 20 field samples or designated sample batch may be performed as specified in the client site plan.

12.2 **Method Blank**

12.2.1 With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For water samples, an organic-free reagent water blank is processed. The method blank must exhibit PCB levels less than the matrix defined reporting limit (RL). If the method blank exhibits PCB contamination above the reportable RL, the samples associated with the contaminated blank should be re-extracted and re-analyzed. If there is no original sample available for re-extraction then the results should be flagged with a "B" indicating blank contamination. The value measured in the blank is reported for those samples associated with the particular blank out of criteria.

12.3 **Laboratory Control Spike**

12.3.1 A Laboratory Control Spike (LCS), also referred to as a QC reference check standard, is extracted with each batch of samples at a rate of one per 20 samples. For a water sample, spike one liter of laboratory organic free water, extract, and analyze. For solid and tissue samples, spike 10 grams of sodium sulfate, extract, and analyze. For oil samples, spike 1 gram of PCB free oil, extract, and analyze. An Aroclor

is chosen for the LCS analyte, typically based on program requirements or expected sample contamination. Calculate the percent recovery for the PCB spike. If the percent recovery for the LCS is out of the acceptance criteria of 70%-130%, the analysis is out of the control, and the problem should be immediately corrected.

12.3.2 The following are default Laboratory Spikes Concentrations:

Aqueous Samples: 1.0 mL of A1242 @ 0.5 ug/mL (ppm) yielding a final sample concentration of 0.500 ug/L.

Solid Samples: 1.0 mL of A1242 @ 100 ug/mL (ppm) yielding a final sample concentration of 10 ug/g.

Note: Alternate spike concentrations and selection of Aroclors may be applicable based on project specific requirements.

12.4 **Duplicate Analysis**

12.4.1 Duplicate analysis of the same sample is performed to assess method precision. A duplicate can also be performed as a blind duplicate, so that identification with original sample is withheld. The analysis of a duplicate sample precludes that PCBs are to be found at appreciable levels in samples. If this is not known, the analysis of a matrix spike or matrix spike duplicate provide more consistent quality control information. The relative percent difference of the two measurements on the sample is calculated on total PCB concentration by the following equation:

RPD = (DUP1-DUP2)/AVG x 100

Where: RPD = Relative Percent Difference
DUP1 = The greater of the measured values
DUP2 = The lesser of the measured values
AVG = Average of the two analysis

The relative percent difference must be $\leq$ 30%.

12.5 **Matrix Spike and Matrix Spike Duplicate (MS/MSD)**

12.5.1 A matrix spike is to be analyzed at a rate of one matrix spike per every 20 samples. Also matrix spike duplicate or duplicate sample is to be analyzed at a rate of one per every 20 samples. Duplicate samples may be appropriate in place of matrix spike duplicate, for soil and waste samples, where detectable amounts of organics are present.

12.5.2 The following are default Laboratory Matrix Spike Concentrations:

Aqueous Samples: .1.0 mL of A1242 @ 0.5 ug/mL (ppm) yielding a final sample added concentration of 0.500 ug/L

Solid Samples: 1.0 mL of A1242 @ 100 ug/mL (ppm) yielding a final sample added concentration of 10 ug/g .

Note: Alternate spike concentrations and selection of Aroclors may be applicable based on project specific requirements.

12.5.3 Analyze one unspiked and one spiked sample. Calculate the percent recovery based on PCB concentration of both samples as follow:

P = A-B/T x 100

Where: P = Percent recovery, %
A = Concentration of analyte in the spike sample aliquot
T = Know true values of the spike concentration
B = Background concentration of PCB in the unspiked sample aliquot

12.5.4 Matrix spike recovery information is used to assess the long-term precision and accuracy of the method for each encountered matrix. Matrix spike/matrix spike duplicate results are not used alone to qualify an extraction batch. Generally, percent recovery for MS/MSD samples should have acceptance limits of $\geq$ 70% and $\leq$ 130%, based on the total PCB concentration. If the percent recovery is outside the acceptance limits, all calculations should be checked and the data should be narrated to describe possible matrix interference.

12.6 Surrogates

12.6.1 A surrogate compound is added to each sample, matrix spike, matrix spike duplicate, duplicate, method blank, and LCS at the time of extraction. The surrogate compounds chosen for this method are Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP). The following are typical surrogate amounts added to normal encountered matrices. These amounts can be adjusted if the PCB background levels are high, and the surrogate is outside its acceptance range.

12.6.1.1 Soil, sediment, sludge, oil, fuel oil, waste solvent, fish, other aquatic animals, tissue samples: 0.5ml of 0.5ppm TCMX/ 5.0ppm DCBP set to 25ml final extract volume.

12.6.1.2 Water: 1.0ml of 0.05ppm TCMX/ 0.5ppm DCBP; set to 10ml final extract volume.

12.6.2 Only one surrogate analyte needs to meet established control limits for the analysis to be valid. The recovery must fall within lab established default limits of 60-140% unless otherwise noted. If percent surrogate recovery is not within laboratory established limits for either surrogate, the following steps are required.

12.6.2.1 Review calculations that were used to generated surrogate percent recovery values to make certain there are no errors.

12.6.2.2 Check by GC analysis surrogate solutions used during sample extraction steps to ensure that no problems exist with spiking solutions.

12.6.2.3 Review data for chromatographic interferences.

12.6.2.4 Re-extraction and/or re-analysis of samples may be indicated if problems persist with surrogate recoveries. If the surrogate percent recovery is out of limits on the re-extracted samples, low or high surrogate recovery is due to matrix affects and the data can be reported as estimated. If the above steps do not lead to satisfactory results then consult with the Operations Manager to resolve the situation.

12.7 Continuing Calibration Check Standard (CCCS)

12.7.1 The initial CCCS is from an alternative source independent of the calibration check standards. It is prepared at a concentration approximately equal to the midlevel standard. This standard is analyzed after the initial calibration standards, every tenth injection, and at the end an analytical sequence. One check standards must be run within a 24-hour time period. The percent recovery must be $\pm$15% of the true value.

12.7.2 If the criterion is exceeded, the analyst should inspect the system to determine the cause and perform maintenance as necessary. The system can then be recalibrated and sample analysis can proceed. Note that all samples which are not bracketed by valid check standards must be re-analyzed when the system is in-control.

12.8 Retention Time

12.8.1 The retention time (RT) windows are established from the Continuing Calibration Check Standard (CCS) peak retention times. The CCS is analyzed three times over a 72-hour period and the standard deviation is calculated from the three retention time measurements. The standard deviation (SD) is multiplied by three and this establishes the retention time window for each quantified peak (±3 SD). Use the retention time for a peak in the continuing calibration check standard to determine the mid-point of the retention time window for the analysis sequence. If the continuing calibration checks fall outside of these windows, update the windows using the previous check standard. If the retention times are still outside the established windows, instrument maintenance must be performed and recalibration may be required.

12.8.2 This function is performed in the chromatography software, shown graphically, as vertical dropdown retention time markers with retention time window brackets. Besides evaluating the retention time windows in order to assign peaks for quantification, the analyst should also rely on their experience in pattern recognition of multi-response sample analysis.

12.8.2 See attachment E for an example of calculated retention time windows. Refer to the Retention Time Window Study for GC18F Column DB-1.

12.9 Analytical Sequence Queue:

12.9.1 The following is an example of the order that initial calibration standards, continuing calibration check standards, method blanks, QC samples, and samples are placed in an analytical sequence. A continuing calibration check standard is run after every ten samples in the analytical sequence. All analytical sequences must end with a continuing calibration check standard regardless of the number of samples. Below is an example of an analytical sequence.

Injection Number	Injected Sample
1	Hexane Blank
2-36	Initial Calibration Standards
37-43	Continuing Calibration Check Standard
44-52	Samples analyses, including method blanks, matrix spikes, matrix duplicates, and QC reference check standards. A maximum of 9 samples between continuing calibration check standards.
53	Continuing calibration check standard
54+	Repeat injection. 44-53 sequence

12.10 PCB Aroclor Qualitative Identification and Secondary GC Column Confirmation:

12.10.1 Positive identification of PCB Aroclors is based on comparison of retention time of the five selected quantitation peaks and major non-quantitation peaks for the unknown sample with retention time of reference standards (continuing calibration verification standards). Additionally pattern recognition is used for comparison of unknown samples with reference standards for positive identification. Confirmation of Aroclor presence by secondary GC column analysis may be necessary for highly altered and/or degraded PCB patterns for programs including PCB air monitoring, US-EPA CLP protocol, and other projects as specified in the site sampling and analysis Quality Assurance Plan (QAP).

12.10.1.1 Dual Column/Confirmatory Column Analysis by GC:

Inject samples under same operating conditions and analytical run QA/QC parameters on a secondary GC column of dissimilar phase (e.g ZB-1 and ZB-5). Note: If using dual GC column system, samples are injected sequentially through separate injection ports onto both columns. Samples are analyzed and concentration results are reported.

12.10.1.2 Dual Column/Confirmatory Column Laboratory Default by SW-846:

12.10.1.2. 1 Report **highest** concentration of the 2 column results for each individual Aroclor on the merged EDD, Form 1, or Certificate of Analysis (Note: This is appropriate for Aroclor regulated projects (e.g. Air Monitoring for EPA TO-10A alternative reporting may be based upon total PCB values for PCB- Total regulated projects).

12.10.1.2.2 If **RPD percent** exceeds 40%, report the **highest** concentration result of the two analyses unless observed chromatographic interference or instrumental analysis. QA/QC indicates the **lower** value may be more accurate. P-flag all deviations >40% and describe interferences or rationale for reporting lower value in Data Narrative.

12.10.1.2.3 If a concentration is above the PQL on one column and below the PQL on the second column, the qualitative presence is not confirmed and the sample is reported as not detected. **Note: If reporting to the MDL is required do the following:**

For reporting the MDL:

a) If one result is greater than the PQL and other result is < PQL (J-flag) report the **highest** result as confirmed (*unless interference or QC reasons indicate lower value*).

b) If one result is above MDL (J-Flag) and second is Not Detected, report the concentration as **not detected**. (Presence not confirmed).

c) If both results are J-Flag values (< PQL), report the **highest** value of the two.

12.10.1.3 **USEPA-CLP/ASP Program Protocols**

a) Report **Lowest** Value of the two column results for each individual Aroclor on the merged EDD, Form 1, or Certificate of Analysis (Note: This is appropriate for Aroclor regulated projects (e.g. Air Monitoring for EPA TO-10A alternative reporting may be based upon total PCB values for PCB- Total regulated projects).

b) If **Percent Difference** (not RPD%) exceeds 25%, then P-Flag all excursions >25%. Note any chromatographic interferences present in Case Narrative.

c) If one result is >PQL and other result is < PQL (J-Flag) Report the **lowest** result (J-Flagged) value (*confirmed hit*).

d) If one result is above MDL (J-Flag) and second is not detected report the concentration as **not detected** (*presence not confirmed*).

13.0 Calibration and Standardization

13.1 Gas chromatographic operation parameters: See Attachment C

13.2 Initial GC Calibration

13.2.1 GC calibration is performed by the internal standard calibration procedure. Prior to running samples the system must be calibrated and system performance must be verified.

13.2.2 Establish the gas chromatographic operating parameters outlined in the Procedure section and prepare the calibration standards at the five concentrations outlined in the Reagent and Standard section. Inject each calibration standard using the GC Autosampler and the parameters outlined in the Procedure section. Note: The same parameters are used for actual samples.

13.2.3 For each Aroclor, 5 peaks are selected to prepare calibration curves. The peaks selected from the multi-component Aroclor formulations were based on maximizing the separation for each Aroclor (i.e., minimizing peak overlap in retention time). Consideration was also given to selecting peaks that normally did not have problems with co-elution with interfering peaks or possible co-elution with organochlorine pesticides. The determined area of the five peaks selected for calibration is processed by the data workstation as a group, combining the area for calculations of the calibration factors. The following table lists the Aroclors that are included in the initial calibration and the peak numbers used.

Aroclor	Peak Numbers
A1016	6,7,8,9,10
A1221	1,2,3,4,5
A1232	5,7,8,9,10
A1242	6,7,8,9,10
A1248	11,12,13,14,15
A1254	16,17,18,19,20
A1260	20,21,22,23,24

13.2.4 For the initial calibration curve to be considered valid, the percent relative standard deviation of response factors must be < 20% over the working range if the average calibration factor quantitation is used. Note: the % RSD is a useful check for linearity through the origin and is used as a data quality indicator. In general an inverse weighted linear calibration curve with intercept is used for quantitation and is not replaced with the average calibration factor. For linear calibration curve the Correlation Coefficient R must be >0.99.

13.3.5 Our laboratory uses a computer based chromatography software module (Water Corporation, Empower software) interfaced to the gas chromatograph. The workstation processes the detector signal, performs an analog to digital conversion, and stores the digitized chromatograms on the computer hard disk. Integration of peak areas, production of chromatograms, calculating calibration curves/response factors, report generation, and archival of data, are all performed in the Empower software. All data results will then be transferred to specialized software developed at Northeast Analytical called NEA LIMS. Once results for calibration curves are transferred to NEA LIMS, they are then saved and set for association with subsequent samples sets.

13.3.6 If a re-calibration is performed, the CCCS must be analyzed again, and values calculated use the new relative response factors. If the CCCS fails to meet the percent difference criteria after re-calibration, sample analysis must not proceed until the problem is found and corrected (*i.e.*, GC gas leak, autosampler lines plugged, broken injector liner).

13.3 Retention Time Windows

13.3.1 The GC system should be checked by the analyst to make sure it is functioning properly before establishing retention time windows. Select a calibration standard and inject three times within a 72-hour time period.

13.3.2 For each peak calculate the standard deviation resulting from the variation in the three retention times for that peak.

13.3.3 The retention time window is defined as $\pm$ 3X SD of the three retention time determinations.

13.3.4 If the standard deviation of the selected peak is zero, the standard deviation of the following eluting peak is used. If it is the last eluting peak is zero for the standard deviation, then substitute the standard deviation of the peak eluting before the last peak.

14.0 Procedure

14.1 Sample Extraction and Preparation

14.1.1 The following SOP's detail sample extraction procedures that are utilized in preparing samples for analysis by this analytical method:

SOP NAME	TITLE	EPA Method
NE005	Soxhlet Extraction/ Extract Prep	8082, 3540C
NE017	Fish and Biota Extraction	8082
NE088	PCB Extraction Of Wipe	8082, 3540A
NE 111	Waste Dilution EPA 3580 for PCB 8082	8082, 3580A
NE120	Extraction and cleanup of PCB by SW-846 3550B	8082, 3550B
NE140	PCB Screening by GC	3510C, 3520C, 3545
NE141	SW-846 3510C H2O PCB extraction	8082, 608, 3510C
NE143	EPA 3545 extraction for 8082 PCB	8082, 3545
NE144	EPA 3545 extraction of wipe for 8082 PCB	8082, 3545
NE151	PUF Extraction for 8082 analysis	8082, 3540C/ TO-10A
NE158	% Lipid Determination Fish & Biota	3540, 3500, 2500A
NE194	High Level PCB extraction for 8082 by SepFunnel	8082, 3510C

14.2 Gas Chromatographic Procedures

14.2.1 Prescreening of sample extracts: See standard operating procedure NE140 for details on the PCB screening procedures used prior to final analysis by this method. The GC is standardized by using Aroclor 1221, Aroclor 1242, Aroclor 1260, and surrogate compounds Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP). These three Aroclor formulations incorporate most environmental PCBs found in sample extracts and provide a good estimate of PCB amounts for final dilutions for this determinative method. A three level calibration curve is utilized; Aroclors (0.50ug/ml, 2.5ug/ml, and 5.0ug/ml standards) and surrogates (1/10 ng/ml, 5/50 ng/ml, and 10/100 ng/ml). The concentration of each Aroclor (grouped as Aroclor 1221, Aroclor 1242, and Aroclor 1260 only) in a sample will be calculated based on the extract volume (not the sample weight or volume)

which incorporates solution concentration values that demonstrate if the extract requires dilution for final capillary GC analysis. If a dilution is necessary, sample extracts are diluted to a solution concentration near 0.500ug/ml, which ensures each sample is quantified in the middle of the calibration curve. Prescreening is also used to inspect for method blank contamination, as well as checking surrogate levels and spike concentrations to ensure they remain within QC acceptance limits. In any of the above scenarios, the samples in question are included in a case narrative and then re-extracted.

14.2.2 Approximately 1.0ml of the final dilution extract is transferred into a labeled autosampler vial.

14.2.3 The sequence of the analytical queue is set up in NEA LIMS as a unique batch file. This file contains the exact order in which standards, instrument blanks, and samples will be analyzed. Once the sample set is uploaded into the Empower acquisition/run screen and is saved, the sample set is printed, and the samples are loaded into the GC autosampler tray in the order specified by the sample set queue.

14.2.4 The following labeling will be used on the autosampler vial and for the sample set file created for the analytical queue.

14.2.4.1 The initial calibration standard will be labeled as 040516A, 040516B, etc. Substitute the actual date of analysis and the Aroclor used in the file name.

14.2.4.2 The instrument blanks will be labeled 070405B01, B02, B03, etc. Substitute the actual date of analysis in the file name.

14.2.4.3 The Continuing calibration check standards will be labeled CS160405A CS160405B, etc. Substitute the actual date of analysis and the Aroclor used in the file name.

14.2.4.4 Samples are labeled with the laboratory identification number on the autosampler vial. In the sample set file the laboratory identification number, along with the client identification, sample weight, set volume and dilution are entered.

14.2.5 At this instant, the chromatography software can be initiated to start data collection. The gas chromatograph is placed into run mode and sample analysis is performed until the analytical queue is complete.

14.2.6 Peak Identification

14.2.6.1 Target peaks are identified in unknown samples based upon Retention Time (RT). The retention time of an unknown peak must remain within the retention time windows established.

14.2.6.2 Besides using retention time windows to assign peak IDs, the analyst should also rely on their own experience in recognition of multi-response PCB chromatograms. Caution should be exercised when identifying peaks which elute near interferences present in samples and blanks. Comparison of sample chromatograms with method blank and field blank chromatograms is useful in determining chromatographic interferences.

14.2.6.3 This method should be applied with caution when used in determining PCB of interest in unknown samples for which no prior historical information exists. In this case confirmatory column analysis or confirmation by GC/MS analysis may be advised.

14.3 Data Reduction/Reporting

14.3.1 Final peak assignments and quantitation calculations are performed within the software along with the current instrument calibration. The final concentration results are provided in the reporting section of the software. Final concentration results are reviewed by the QA department or other approved manager before release to the client.

14.3.2 Data Qualifiers:

Sample Concentration Reports (Certificates of Analysis, Data Package Form 1's, and Electronic Data Deliverables (EDDs) are generated using the appropriate data qualifiers as follows:

U – Denotes analyte not detected at concentration ≥ Practical Quantitation Limit (PQL). Note: PQLs are adjusted for sample weight/volume and dilution factors.

J - Denotes an estimated concentration. The concentration result is ≥ Method Detection Limit (MDL) but < Practical Quantitation Limit (PQL).

P - Indicates relative percent difference between primary and secondary GC column analysis exceeds 40 %.

C- Denotes analyte confirmed by secondary GC column analysis.

B - Denotes analyte observed in associated method blank. Analyte concentration should be considered as estimated.

E - Denotes analyte concentration exceeded calibration range of instrument. Sample could not be re-analyzed at secondary dilution due to insufficient sample amount, quick turn-around request, sample matrix interference or hold time excursion. Concentration result should be considered as estimated.

Z - Laboratory Reserved Qualifier (explained in associated Case Narrative).

15.0 Calculations

15.1 Calibration curve calculation:

15.1.1 PCB Solution concentration calculation from initial Calibration by Linear Regression **Yi= aXi + b.**

Xi = Calibrated Solution Concentration (ng/mL)
Yi = total area response of 5 PCB quant. peaks (uV-Sec.)
a = slope
b = intercept

Unknown Solution Conc. **X = (Y –b) a**

Y = Total area response of PCB Chromatogram (uV-Sec.)
a = slope of ICAL by linear regression
b = intercept of ICAL by linear regression

15.2 Capillary GC: Sample calculations

15.2.1 The concentration of each identified PCB Aroclor in a sample will be calculated based on the sample weight or volume.

15.2.2 The PCB solution concentration of the extract is calculated as follows:

Solution Conc. = (Y –b)/a

Where:

Y = Total area response of PCB Chromatogram (uV-Sec.)
a = slope of ICAL by linear regression
b = intercept of ICAL by linear regression

15.3 Final concentration of samples:

15.3.1 Calculations of final PCB concentrations will vary upon matrix, calculations are as follows:

(1.) Soil/Sediment/Solids:

Final Conc. = (Sol. Conc.) * (V)*DF/ (M)* (%Total Solids) (1/1000) ug/g

Where: Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
M = mass extracted (g)

(2.) Water:

Final Conc.= (Sol. Conc.) * V*DF/[(Vt)](1/1000) ug/L

Where: Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
Vt= Total Volume Extraction (L)

(3.) Biota Tissue

Final Conc. = (Sol. Conc.) * (V)*DF/(M)(1/1000) ug/g

Where: Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
M = mass extracted (g)

(4.) PUF Cassette

Final Conc. = (Sol. Conc.) * (V)*DF/ (Va) ng/cubic meter

Where: Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
Va = volume of air sampled (cubic meters)

(5.) Waste Oil

Final Conc. = (Sol. Conc.) * (V)*DF/ (M)*(%Total Solids) (1/1000) ug/g

Where: Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
M = mass extracted (g)

15.3 The calculated concentration for each PCB Aroclor will be compared to its respective sample-specific reporting limit (RL) and method detection limit (MDL). The results with concentrations at or above the MDL, but below RL will be reported as detects and flagged as estimated, J. The results for peaks with concentrations at or above the RL, would be reported as unqualified numeric values.

16.0 Method Performance

Note: Method Performance is Assessed by Initial/Continuing Demonstration of Proficiency Studies and MDL Determinations

16.1 Initial Demonstration of Performance (IDOP) Procedure:

16.1.1 Prepare 4 replicates of a fortified laboratory blank sample (using laboratory reagent water or sodium sulfate) by spiking each sample with 1.0 mL of 0.500 ug/mL Aroclor solution (typically Aroclor 1242) for water samples

and 0.100 mL of 100 ug/mL Aroclor 1242 solution for solid samples. Prepare one method blank sample with the batch. Extract and analyze each aliquot according to procedures beginning in Section 14.0 below.

16.1.2 For each replicate the recovery value of the sample must remain in the acceptance range between 70 – 130% (or established lab limits) and the percent RSD must be < 20 % for the method performance to be considered acceptable.

16.1.3 This procedure must be repeated using four fresh samples until satisfactory performance has been demonstrated. The initial demonstration of capability is used primarily to preclude the laboratory from analyzing unknown samples via a new, unfamiliar method prior to obtaining some experience with it.

16.2 Continuing Demonstration of Performance Procedure:

16.2.1 Annual continuing demonstration of performance may be satisfied by an Initial Demonstration of Performance (IDOL). The IDOL can consist of an acceptable analysis of unknown samples (for example PT test sample), or the acceptable analysis of 4 consecutive Laboratory Control Spike samples. Records of continuing demonstration of performance are maintained by the laboratory Quality Assurance Department.

16.2.2 With each batch of samples to be extracted, a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For water samples, an organic-free reagent water blank is processed.

16.2.3 The method blank should exhibit PCB levels less than the practical quantification limit or reporting limt (PQL or RL). If the method blank exhibits PCB contamination above the reportable quantitation limit, the samples associated with the contaminated blank should be re-extracted and re-analysis when appropriate. If the original sample is not available for re-extraction or if the associated sample concentrations greatly exceed the blank concentration, then all positive concentration results for the associated samples should be flagged with a "B", indicating blank contamination and a Case Narrative describing the situation should be prepared.

16.2.4 A matrix spike is analyzed at a rate of 1 matrix spike per every 10 samples. A duplicate sample may be prepared in lieu of a matrix spike when detectable PCB concentrations are known to be present.

16.3 Method Detection Limit

16.3.1 A method detection limit will be determined for this method whenever major modification to the extraction or analysis procedures are made or at a minimum frequency of every 2 years. A minimum of seven laboratory organic free water samples or sodium sulfate samples will be prepared and spiked with chlorinated PCB methyl esters mixture, at a low level, and taken through all extraction and analytical procedures.

$$\mathbf{MDL = S * t_{(n-1,\ 1\text{-}alpha=0.99)}}$$

Where:

S = Standard deviation of the replicate analyses
n = Number of replicates
$t_{(n-1,\ 1\text{-}alpha=0.99)}$ = Student's t value for the 99% confidence level with n-1

For example: t for 8 replicates = $t_{(7,0.99)}$ = 2.998

16.3.2 The determined MDL must be less than the concentration spiked but greater than one tenth (1/10) the spiked concentration. If not, repeat the MDL determination at an appropriate spike concentration for affected analytes.

17.0 Pollution Prevention

17.1 Pollution prevention is practiced in the laboratory by minimizing usage of solvents and chemicals, so that disposal of waste generated is held to the smallest amount possible. This is directly linked to the types of extraction procedures in place at the laboratory to reduce the volumes of solvents used for semi-volatile extraction procedures. Northeast

Analytical employs extraction procedures such as continuous liquid/liquid and solid phase extraction methods to reduce solvent requirements for water extraction protocols and ASE and Soxhlet extractions for solid matrices.

17.2 Pollution prevention also relies on minimizing the chemicals and solvents required to perform extraction and analysis procedures. NEA laboratory personnel strive to purchase chemicals and standards that will be consumed based on anticipated workload. For additional information about laboratory pollution prevention, please refer to laboratory SOP NE168.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 The GC analyst is responsible for generating the data and the initial review of the data. This would include inspection of the chromatographic data, processing the raw data, producing all required data forms, inspection of calibration curves for compliance, surrogate recovery, laboratory control spike recovery, matrix spike/matrix spike duplicate recovery, and continuing calibration compliance.

18.2 Once the initial review of the data is performed by the analyst, decisions are made at that time to accept the data if all criteria are met or to reject sample data if any of the quality control parameters or limits are out of control. Depending on the situation, samples requiring re-extraction will be notified to the appropriate extraction personnel, sample extracts requiring re-injection will be queued for analysis, new calibrations may have to be performed, or samples re-analyzed due to failing continuing check standards.

18.3 The analyst may also consult with the quality control officer as to the best form of action to take or if the situation warrants corrective action beyond routine practices. If no recourse is available, and the data is to be reported out of criteria, a Case Narrative Report is generated and the deviation is documented and reported to the client. The Case Narrative Report is filed with the data and is also useful for production of case narratives that are issued with the final data reports. If a problem exists that requires follow-up to rectify, a Corrective Action Report (CAR) is issued to document the problem found, steps taken to resolve the problem, and document samples that were affected. This CAR form is filed by the Quality Control Officer and reviewed by management to verify that appropriate actions have been taken to correct the problem.

18.4 Please see Table 19.1 below for specific Quality Assurance Acceptance Criteria.

19.0 Corrective Action for Out-Of-Control Data

19.1 The table below outlines the data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

Quality Control Acceptance Criteria and Corrective Action Plan

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	The five point calibration is analyzed initially and when Continuing Calibration Check standard fails criteria.	• %RSD ≤20% for the relative response factors for the calibration standards if using average response factor calibration. Correlation Coefficient (R) must be >0.99 for Linear Regression.	• Re-analyze the initial calibration standard and/or evaluate/correct instrument malfunction to obtain initial calibration and continuing calibration check standards that meet criteria.
Continuing Calibration Check Standard (CCCS)	• Initially analyze a CCCS immediately following an initial calibration. • After the initial CCCS of the sequence, a CCCS must be analyzed after 9 samples. • Analytical sequence must end with analysis of a CCCS.	• Calibration factor for the continuing calibration check must be ±15% of the true value. • Retention time of all quantitated peaks must be within RT window (reset with each initial CCCS of a sequence). • All samples must be bracketed by a CCCS that meet all criteria stated above.	• If the reason for the failure of the CCCS appears to be a poor injection (or a degraded standard solution), the CCCS will be re-injected (or re-prepared and re-injected) immediately following the failed CCCS. This can only occur if the instrument is being attended by an analyst. If upon re-injection, the CCCS meets all the acceptance criteria and there is no apparent impact on the sample data, the analytical sequence will continue and samples will not be re-

			analyzed. The associated sample data will be reported. • If CCCS failure was not due to a poor injection (or degraded standard solution) or the instrument was unattended at the time of the CCCS failure, correct system, if necessary, and recalibrate. Initial calibration and CCS criteria must be met before sample analysis may begin. Samples that are not bracketed by complaint CCCSs must be reanalyzed. • If acceptable CCCSs are observed later in the sequence, samples bracketed by acceptable CCCSs will be reported. Samples between the failed CCS and prior/ subsequent complaint CCCS will be re-analyzed.
Retention Time (RT)	• Use the retention time for peak in the CCSs to determine midpoint of the relative retention time window for the analysis sequence. •Each sample analysis: Rely on RT windows to identify PCB Aroclor to report. Also use pattern recognition and professional judgment for peaks that shift from RT windows, because compound composition may shift RT for GC peaks.	• Each quantified peak and surrogate peak should be within established windows.	• Inspect chromatographic system for malfunction, correct problem. Perform re-analysis if necessary.
Method Blank	• One per extraction batch of ≤20 samples of the same matrix per day. • Must be analyzed on each instrument used to analyze associated samples. • Must undergo all sample preparative procedures.	• Concentration does not exceed the RL for any PCB Aroclor. •Must meet surrogate criteria of 60-140% recovery.	• Re-analyze method blank to determine if instrument contamination was the cause. If method blank re-analysis passes, then report samples. • If method blank is found to contain PCB contamination above the RL for any PCB Aroclor compound , then re-extract and re-analyze all associated samples. If no sample exists for re-extraction, report data B-flagged to indicate method blank contamination.
Laboratory Control Spike (LCS)	• One per extraction batch of ≤20 samples per matrix per day.	• Percent recovery must be within method limits. • Must meet Aroclor spike criteria of 70-130% recovery. • Must meet surrogate criteria of 60-140% recovery.	• Re-analyze LCS to determine if instrument was the cause. If LCS passes, then report samples. • If LCS recovery is still out of limits, the re-extract and re-analyze all associated samples. If no sample exists for re-extraction, report data flagged to indicate LCS failed recovery.

Matrix Spike/Matrix Spike Duplicate (MS/MSD)	• Normal method procedure is to extract and analyze a matrix spike sample. One MS per extraction batch of ≤20 samples per matrix per day. • If requested, an MSD can be extracted and analyzed. The MSD would follow the above criteria as for the MS.	• Percent recovery for MS must be within method limits. • If MS/MSD is analyzed, relative percent difference (RPD) should be within 30%. • Must meet Aroclor spike criteria of 70-130% recovery. •Must meet surrogate criteria of 60-140% (unless original unspiked sample is also outside of criteria)	• Re-analyze MS and/or MSD to determine if instrument was the cause. If MS and/or MSD pass, then report samples. • Check for errors such as calculations and spike preparation. • Check original unspiked sample results and surrogate recovery for indications of matrix effects. • If no errors are found, and the associated LCS is within limits, then sample matrix effects are likely the cause. Note exceedance in case narrative.
Surrogates	• Surrogates are added to all samples and QC samples. Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) solution.	• Percent recovery for the surrogate should be 60-140%.	• Re-analyze the affected sample or QC sample to determine if instrument was the cause. If surrogate passes, then report samples. • Check for errors in surrogate calculation and surrogate solutions. • If no problem is found, then re-extract and re-analyze the sample. • If re-extraction is within limits and sample extract holding time, then report only the re-analysis. • If the re-extraction is within limits, but out of extraction holding time, then report both sets of data. •If the re-extraction produces surrogate recovery still out of limits, then report both sets of data. •If no sample exists for re-extraction, report data flagged to indicate surrogate failed recovery or have a client re-sample.

20.0 Contingencies for Handling Out-Of-Control or Unacceptable Data

20.1 Data that is detected to be out-of-control for any reason, when compared to method acceptance criteria, it will be addressed in the following manner:

20.1.1 If the problem exists with the gas chromatographic instrumentation, appropriate action will be taken to repair and perform maintenance to bring the instrument back to operation condition. Once the instrumentation is determined to be operating correctly, analysis can begin again.

20.1.2 If the problem exists with calibration standard solutions, the analyst will prepare new standards and discard the standard solutions that are suspect. Instrument calibration can be performed and analysis can begin once system is in control.

20.1.3 If the problem exists with sample extraction and extract preparation, the extraction step that is producing the out of-control situation will be diagnosed and rectified. Once the troubleshooting procedures correct the problem, extraction can once again occur and analysis can continue.

20.1.4 In situations where data is reported for out-of-control conditions, the data will be annotated with data qualifiers and/or appropriate descriptive comments defining the nature of the excursion in the sample Case Narrative. If warranted, a corrective action report (CAR) will be issued to define the problem, correct the problem, and finalize a resolution.

21.0 Waste Management

21.1 All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste generated during the execution of this method.

21.2 Please refer to standard operating procedures, NE089 and NE054, regarding how hazardous waste is handled and disposed of by the laboratory.

22.0 References:

22.1 U.S. EPA SW-846 "Test Methods for Evaluating Solid waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

22.2 U.S. EPA 40 CFP Part 136, "Guidelines Establishing Test Procedures of the Analysis of Pollutants", July, 1988.

22.3 "Standard Methods for the Examination of Water and Waste Water", 19th Edition 1995, American Public Health Association, American Water Works Association, Water Pollution Control Federation.

22.4 New York State Department of Health, "Environmental Laboratory Approval Program Certification Manual", Wadsworth Center for laboratories and Research, 1996.

22.5 Guide to Environmental Analytical Methods", Third edition, Genium Publishing Corporation, 1997.

23.0 Tables, Diagrams, Flowcharts and Validation Data

Attachment A: PCB Stock Standard/Calibration Standard Preparation

Attachment B: Continuing Calibration Check Standard Preparation

Attachment C: GC Operating Parameters

Attachment D: Chromatograms

Attachment E: Example Calibration Report

Attachment F: Example Retention Time Window Study

Attachment G: Example MDL Studies

Attachment A: PCB Stock Standards Prep Table:

Table 1

PCB Stock Standard Preparation Table

PCB Formulation	Supplier	Catalog #	Standard weight(mg)	Conc. (PPM)
A1016	Monsanto Neat Archive	NA	100.0	1000.0
A1221	Monsanto Neat Archive	NA	100.0	1000.0
A1232	Monsanto Neat Archive	NA	100.0	1000.0
A1242	Monsanto Neat Archive	NA	100.0	1000.0
A1248	AccuStandard	C-248N-50mg	100.0	1000.0
A1254	Monsanto Neat Archive	NA	100.0	1000.0
A1260	Monsanto Neat Archive	NA	100.0	1000.0
TCMX/DCBP (Surrogate)	Ultra Scientific	CUS-4911*	0.5/5.0	500/5000

Unless otherwise noted hexane is the solution used to make all dilutions. *Custom Ordered.

Table 2A

PCB Calibration Standard Preparation Table (High Level Calibration Curve)

Initial Volume (mL)	Initial Conc. (ug/mL)	Final Volume (mL)	Final Concentration (PPM)					
			A1016	A1221	A1232	A1242	A1248	A1260
5.0	(10.0)	50.0	1.000	1.000	1.000	1.000	1.000	1.000
2.5	(10.0)	50.0	0.500	0.500	0.500	0.500	0.500	0.500
1.25	(10.0)	50.0	0.250	0.250	0.250	0.250	0.250	0.250
0.500	(10.0)	50.0	0.100	0.100	0.100	0.100	0.100	0.100
5.0	(0.200)	50.0	0.020	0.020	0.020	0.020	0.020	0.020

Actual Concentration, see Table 1 for actual working standard concentrations for each Aroclor.
See Table 3A for A1254 Standard Preparation (High Level).

Table 2B

PCB Calibration Standard Preparation Table (Low Level Calibration Curve)

Init. Volume (mL)	Initial Conc. (ug/ml)	Final Volume (mL)	Final Concentration (PPM)					
			A1016	A1221	A1232	A1242	A1248	A1260
0.5	(10.0)	50.0	0.100	0.100	0.100	0.100	0.100	0.100
2.5	(1.0)	50.0	0.050	0.050	0.050	0.050	0.050	0.050
1.0	(1.0)	50.0	0.020	0.020	0.020	0.020	0.020	0.020
1.0	(0.500)	50.0	0.010	0.010	0.010	0.010	0.010	0.010
0.50	(0.500)	50.0	0.005	0.005	0.005	0.005	0.005	0.005

Actual Concentration, see Tables 1 and 2 for actual working standard concentrations for each Aroclor.
See Table 3B for A1254 Standard Preparation (Low Level.)

Table 3A

PCB A1254 Calibration Standard Preparation Table (for High Level Curve)

Initial Volume (mL) A1254	Initial Conc. (ug/mL) A1254	Initial Volume (mL) 0.5/5.0 -PPM Surrogate	Final Volume (mL)	Final Concentration (PPM)		
				A1254	TCMX	DCBP
5.0	10.0	0	50	1.000	0	0
2.5	10.0	0	50	0.500	0	0
10.0	10.00	4.0	100	1.000	0.020	0.200
25.0*	1.000	NA	50	0.500	0.010	0.100
1.25	10.0	0.800	50	0.250	0.008	0.080
0.500	10.0	0.500	50	0.100	0.005	0.050
1.000**	1.000	0.200	50	0.020	0.002	0.020

*This initial volume is of the A1254 1.000 ppm calibration standard WITH surrogates.
**This initial volume is of the A1254 1.000 ppm secondary stock solution WITHOUT surrogates.

Table 3B

PCB A1254, TCMX and DCBP Calibration Standard Preparation Table (Low Level Curve)

Initial Volume A1254 (mL)	Initial Conc. A1254 (ug/mL)	Initial Volume (mL) 0.5/5.0 -PPM Surrogate	Final Volume (mL)	Final Concentration (PPM)		
				A1254	TCMX	DCBP
5.00	1.000	0.80	50	0.100	0.00800	0.0800
2.50	1.000	0.50	50	0.050	0.00500	0.0500
1.0	1.000	0.40	50	0.020	0.00400	0.0400
1.0	0.500	0.250	50	0.010	0.00250	0.0250
0.50	0.500	0.100	50	0.005	0.00100	0.0100

ATTACHMENT B: PCB Continuing Calibration Standards

Table 1

PCB Continuing Calibration Working Standards prepared from 1000 PPM Stock Standards

PCB	Stock Source	Initial Volume (mL)	Final Volume (mL)	Concentration (PPM)
A1016	Chem Service Cat # F107AS	1.0	100	10.0
A1221	Chem Service Cat # F108AS	1.0	100	10.0
A1232	Chem Service Cat# F113AS	1.0	100	10.0
A1242	Chem Service Cat# F109AS	1.0	100	10.0
A1248	Chem Service Cat# F110AS	1.0	100	10.0
A1254	Chem Service Cat# F111AS	1.0	100	10.0
A1260	Chem Service Cat# F112AS	1.0	100	10.0

ATTACHMENT B

Table 2

PCB Continuing Calibration Standards (High Level) prepared from 10 PPM CCV Working Standards and Surrogates

PCB	Surr. Volume* (mL)	Spike Volume (mL)	Final Volume (mL)	Surrogate Concentration TCMX/DCBP (PPM)	Aroclor Concentration (PPM)
A1016	2.0	5.0	100	0.010/0.100	0.500
A1221	2.0	5.0	100	0.010/0.100	0.500
A1232	2.0	5.0	100	0.010/0.100	0.500
A1242	2.0	5.0	100	0.010/0.100	0.500
A1248	2.0	5.0	100	0.010/0.100	0.500
A1254	2.0	5.0	100	0.010/0.100	0.500
A1260	2.0	5.0	100	0.010/0.100	0.500

*Surrogate stock solution 0.500 PPM TCMX and 5.0 PPM DCBP

Table 3

PCB Continuing Calibration Standards (Low Level) prepared from 0.500 PPM CCV Standards and Surrogates

PCB	Surr. Volume* (mL)	Initial Volume (mL)	Final Volume (mL)	Surrogate Concentration TCMX/DCBP (PPM)	Aroclor Concentration (PPM)
A1016	.100	1.0	10	0.005/0.050	0.050
A1221	.100	1.0	10	0.005/0.050	0.050
A1232	.100	1.0	10	0.005/0.050	0.050
A1242	.100	1.0	10	0.005/0.050	0.050
A1248	.100	1.0	10	0.005/0.050	0.050
A1254	.100	1.0	10	0.005/0.050	0.050
A1260	.100	1.0	10	0.005/0.050	0.050

*Surrogate stock solution 0.500 PPM TCMX and 5.0 PPM DCBP.

Attachment C: GC Operating Parameters

GC-19 Low Level PCB Method

GC #:	**19**
Method:	**608/8082 Low Level PCB**
GC Method #:	**5**
Date:	**10/27/2006**
Analyst:	**AJM**
File Name:	**S:\FORMS\LOG\GC\[GC19_Parameters.xls]8082 LL M5**
Column:	**19F ZB-1**
	19B ZB-5

Sample Delivery:

Single Method **Method 5**

CP-8400

Injection Mode:	**User**		
Sample Penetration Depth (%)	95	Viscosity Delay (sec)	1.0
Solvent Penetration Depth (%)	95	Plunger Speed During Fill (ul/sec)	1.0
		Plunger Speed During Injection (ul/sec)	5.0
Air Plug after Sample (ul)	1.0	Pre Injection Delay (sec)	3.0
Sample Air Gap	No	Post Injection Delay (sec)	3.0
Number of Fill Strokes	0		
Fill Volume for Fill Strokes	5.0		

Default Clean

Default Clean Vial	I
Default Clean Volume (ul)	5
Number of Clean Strokes	1
Default Clean Drawup Spped (ul/sec)	5

Clean Mode

Number of Pre-Injection Solvent Clean Flushes	1
Number of Post-Injection Solvent Clean Flushes	1
Number of Pre-Injection Sample Clean Flushes	0
Clean Solvent Source Vial	I+II

Solvent Plug

Vial for Solvent Plug	III
Solvent Plug Size (ul)	0.2
Solvent Drawup Speed (ul/sec)	5.0
Solvent Pause Time	1.0
Solvent Air Gap	NO

Select Edit

Select Automation Mode:	Single Method		
Edit Single Method Automation			
Method:	5		
Initial Sample:	0	Injection Possition	Both
Final Sample:	99	First Injector used	Pos. 1
Injections / Sample:	1	Use Injection Delay	No
First Injection Volume (ul):	1.1*	Delay between Injections	0.5
Second Injection Volume (ul):	1.3*	Advance Carrousel between Injections	No
	*can vary	Clean between Injections	Yes

Column Oven:

Step	Temp (°C)	Rate (°C/min)	Hold (min)	Total (min)
Initial	140	-------	2.00	2.00
2	200	10	0.00	8.00
3	245	5	13.23	30.23

Stabilization Time (min): 0.20

Injector: Front CP-1177

1177 Oven Power: ON
1177 Temperature (°C) 300

Time	Split State	Split Ratio
Initial	ON	20

Flow/PSI (Front EFC, Type 1):

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	*23.1**	-------	30.30	30.30

*last adjustment

Constant Flow Mode Enable: NO
Column Flow Rate (ml/min): 1.5

Injector: Middle CP-1177

1177 Oven Power: ON
1177 Temperature (°C) 300

Time	Split State	Split Ratio
Initial	ON	20

Flow/PSI (Front EFC, Type 1):

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	*29.5**	-------	30.30	30.30

Constant Flow Mode Enable: NO
Column Flow Rate (ml/min): 1.5

Detector: **Front ECD**

ECD Oven Power: ON
Temperature (°C) 300
Electronics: ON
Range: 1

Time	Range	Autozero
Initial	1	YES

Front ECD Adjustment
Time Constant: Fast
Cell Current: CAP
Contact Potential (mV): -110
Date of last adjustment 5/2/2006
Make-Up Flow (ml/min): manually set and measured.

Middle ECD

ECD Oven Power: **OFF**
Temperature (°C) 50
Electronics: ON
Range: 1

Time	Range	Autozero
Initial	1	YES

Fast
CAP
-20
5/2/2006

Analog Output

Detectors: Front: ECD
Middle: ECD
Rear: None

Time	Signal Source	Attenuation
Initial	Front Detector	1
Time	Signal Source	Attenuation
Initial	Middle Detector	1
Time	Signal Source	Attenuation
Initial	Rear Detector	1

Valve Table:

Time	1 None	2 None	3 None	4 None	5 None	6 None	7 None
Initial	-----	-----	-----	-----	-----	-----	-----

Initial valve state=Off

GC-19 8082 High Level PCB Method

GC #:	**19**
Method:	**8082 High Level PCB**
GC Method #:	**7**
Date:	**2/15/2006**
Analyst:	**KLL**
File Name:	**S:\FORMS\LOG\GC\[GC19_Parameters.xls]8082 HL M7**
Column:	**19F ZB-1**
	19B ZB-5

Sample Delivery:

Single Method **Method 7**

CP-8400

Injection Mode:	**User**		
Sample Penetration Depth (%)	95	Viscosity Delay (sec)	1.0
Solvent Penetration Depth (%)	95	Plunger Speed During Fill (ul/sec)	1.0
		Plunger Speed During Injection (ul/sec)	5.0
Air Plug after Sample (ul)	1.0	Pre Injection Delay (sec)	3.0
Sample Air Gap	No	Post Injection Delay (sec)	3.0
Number of Fill Strokes	0		
Fill Volume for Fill Strokes	5.0		

Default Clean

Default Clean Vial	I
Default Clean Volume (ul)	5
Number of Clean Strokes	1
Default Clean Drawup Spped (ul/sec)	5

Clean Mode

Number of Pre-Injection Solvent Clean Flushes	1
Number of Post-Injection Solvent Clean Flushes	1
Number of Pre-Injection Sample Clean Flushes	0
Clean Solvent Source Vial	I+II

Solvent Plug

Vial for Solvent Plug	III
Solvent Plug Size (ul)	0.2
Solvent Drawup Speed (ul/sec)	5.0
Solvent Pause Time	1.0
Solvent Air Gap	NO

Select Edit

Select Automation Mode:	Single Method		
Edit Single Method Automation Method:	7		
Initial Sample:	0	Injection Possition	Both
Final Sample:	99	First Injector used	Pos. 1
Injections / Sample:	1	Use Injection Delay	No
First Injection Volume (ul):	1	Delay between Injections	0.5
Second Injection Volume (ul):	1	Advance Carrousel between Injections	No
		Clean between Injections	Yes

Column Oven:

Step	Temp (°C)	Rate (°C/min)	Hold (min)	Total (min)
Initial	140	-------	2.00	2.00
2	200	10	0.00	8.00
3	245	5	13.23	30.23

Stabilization Time (min): 0.20

Injector: Front CP-1177

1177 Oven Power:	ON
1177 Temperature (°C)	300

Time	Split State	Split Ratio
Initial	ON	35

Flow/PSI(Front EFC, Type 1):

Injector: Middle CP-1177

1177 Oven Power:	ON
1177 Temperature (°C)	300

Time	Split State	Split Ratio
Initial	ON	35

Flow/PSI(Front EFC, Type 1):

Table 1: LEAP GC PAL Parameters

Leap GC Pal Parameters 3/9/2007					
Sample injection Methods					
Method	**GC Dual**	**GC Duals**		**Method**	**GC Inj s**
Cylcle	GC Dual	GC Dual		**Cylcle**	GC Inj S
Syringe	10 ul	10 ul		**Syringe**	10 ul
1. Sample Vol	1.0 ul	1.0 ul		**1. Sample Vol**	1.0 ul
1. Air Vol	1.0 ul	1.0 ul		**Solvent Plug**	200 nl
1. Inject to	GC Inj 1	GC Inj 1		**Slv Source**	Standard
Inj Time Diff	0s	0s		**Int Standard**	0 nl
2. Sample Offs	1	0		**Std Source**	Standard
2. Sample Vol	1.0 ul	1.0 ul		**Air Gap (s)**	1.0 ul
2. Air Vol	1.0 ul	1.0 ul		**1. Air Vol Ndl**	1.1 ul
2. Inject to	GC Inj 2	GC Inj 2		**Pre Cln Slv 1**	2
Pre Cln Slv 1	2	2		**Pre Cln Slv 2**	2
Pre Cln Slv 2	2	2		**Fill Speed**	5.0 ul/s
Pre Cln Sp 1	0	0		**Pull Up Delay**	1.0 s
Int Cln Slv 1	2	2		**Inject to**	GC Inj 1
Int Cln Slv 2	2	2		**Inject Speed**	5.0 ul/s
Pst Cln Slv 1	2	2		**Pre Inj Del**	0 ms
Pst Cln Slv 2	2	2		**Pst Inj Del**	0 ms
Fill Volume	10 ul	10 ul		**Pst Cln Slv 1**	2
Fill Speed	2.5 ul/s	2.5 ul/s		**Pst Cln Slv 2**	2
Fill Stroke	0	0			
Pull Up Delay	500ms	500ms			
Inject Speed	10 ul/s	10 ul/s			
Pre Inj Del	0 ms	0 ms			
Pst Inj Del	0 ms	0 ms			

ATTACHMENT D: ZB5 Chromatograms

FIGURE 1. A1016 @ 0.500PPM PLOT

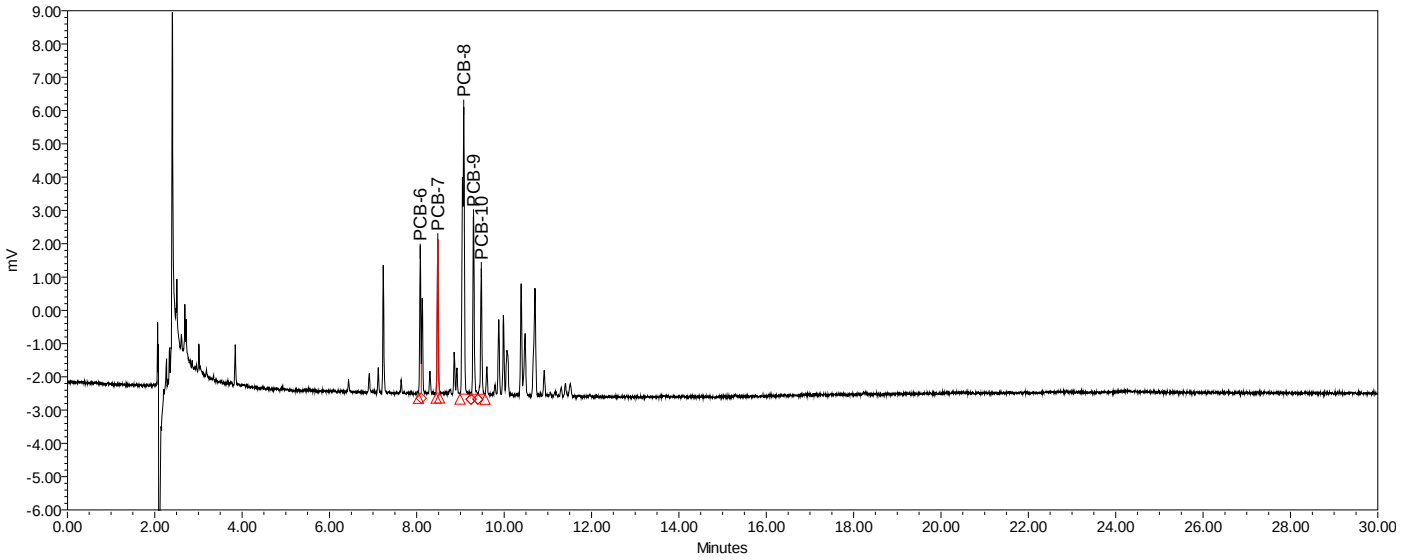


FIGURE 2. A1221 @ 0.500PPM PLOT

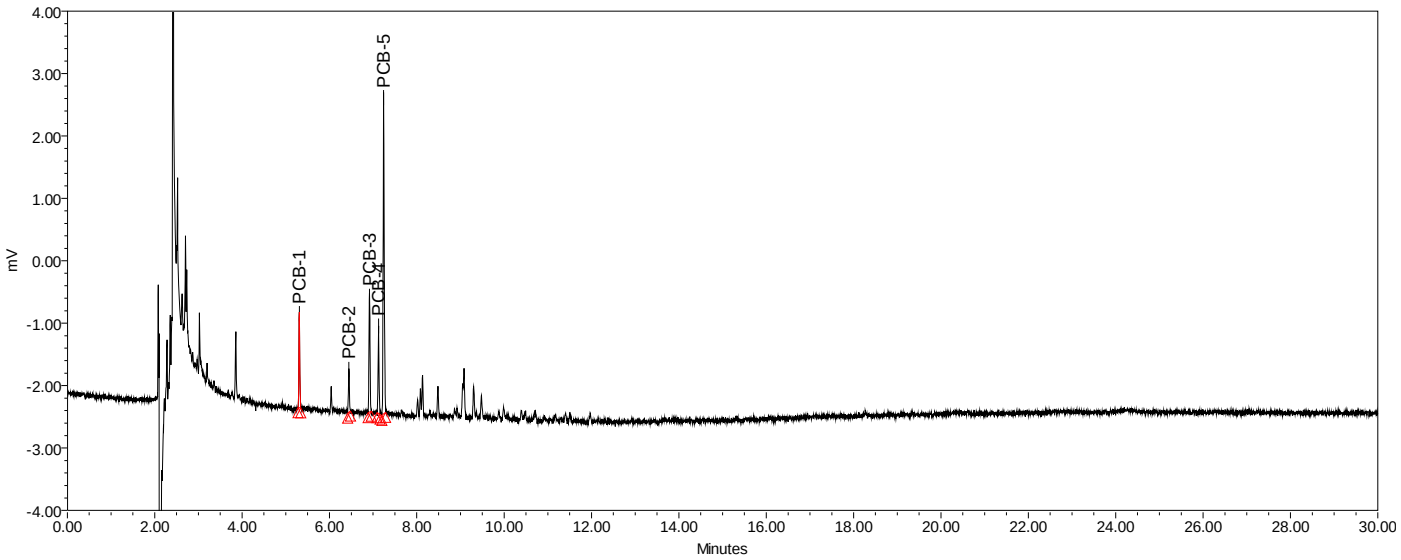


FIGURE 3. A1232 @ 0.500PPM PLOT

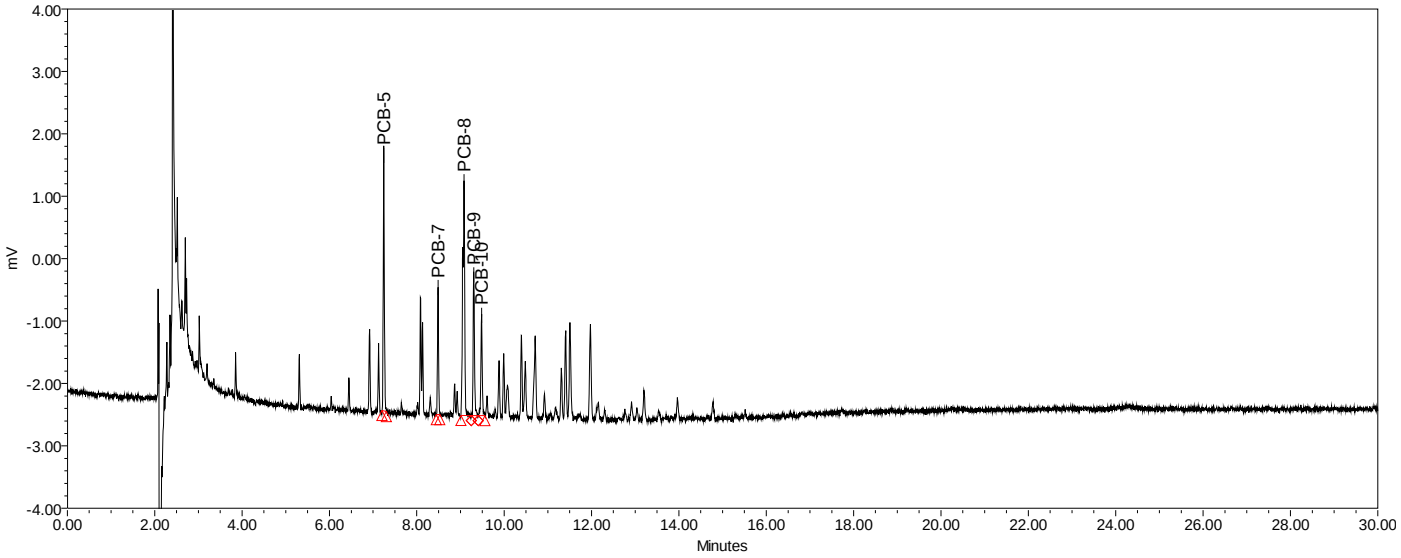


FIGURE 4. A1242 @ 0.500PPM PLOT

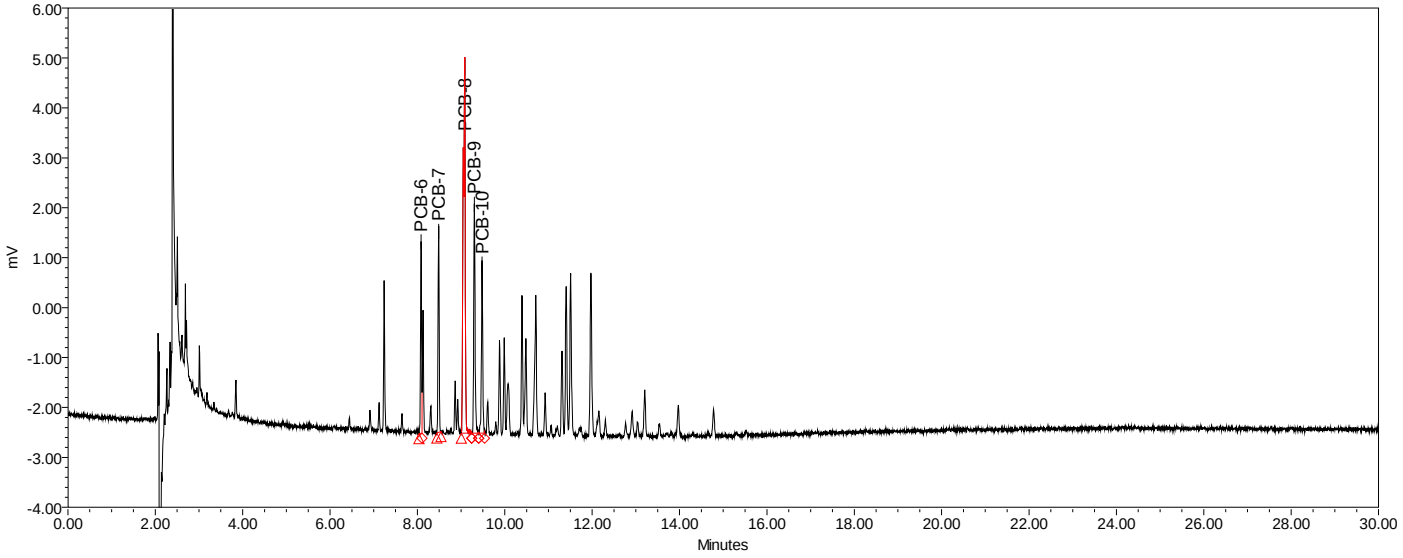


FIGURE 5. A1248 @ 0.500PPM PLOT

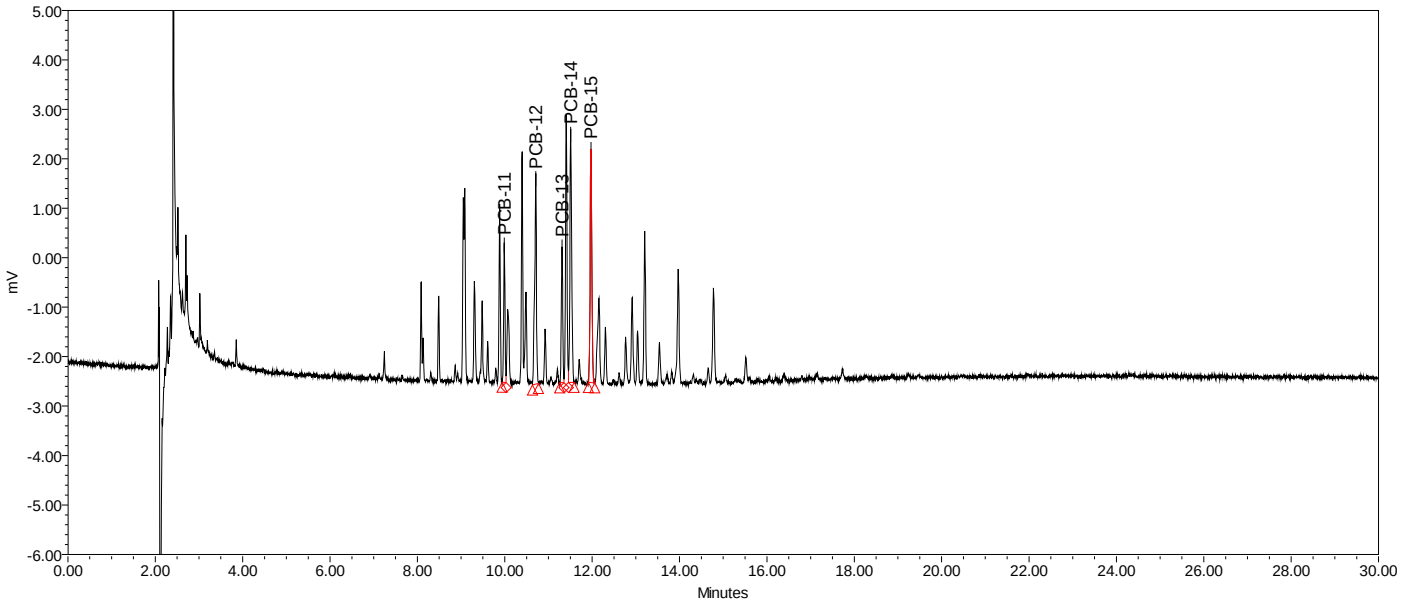


FIGURE 6. A1254 @ 0.500PPM w/ TCMX@10ppb & DCBP@100ppb PLOT

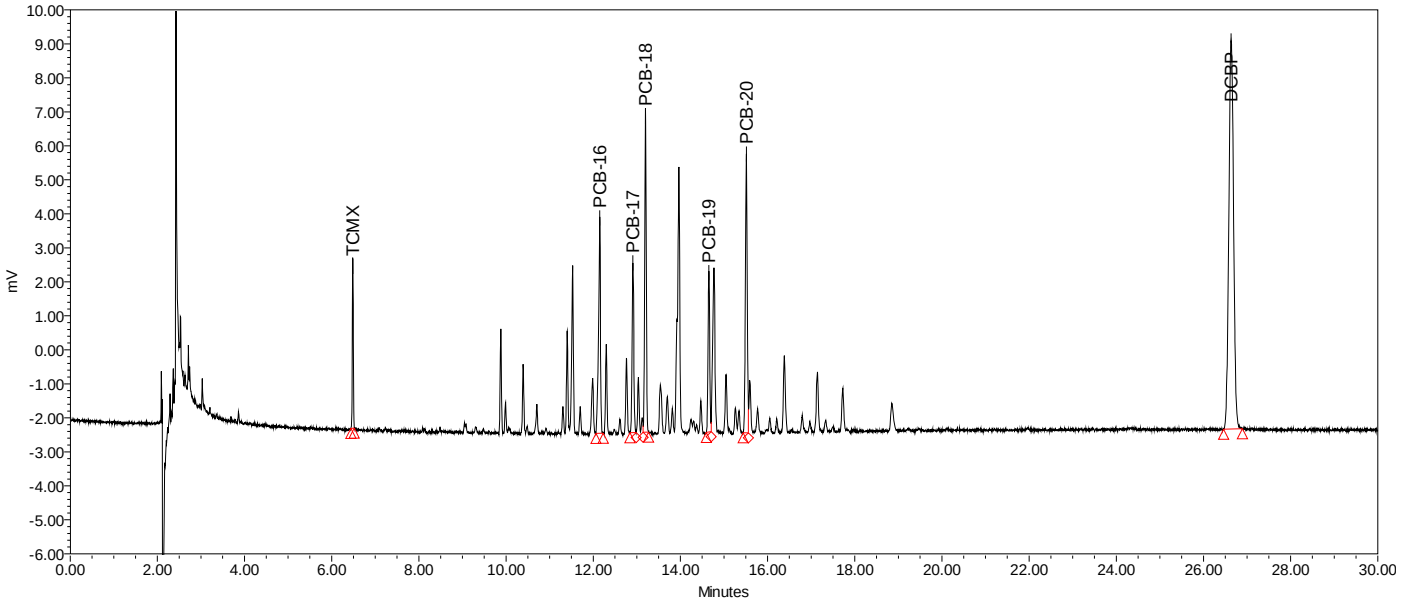
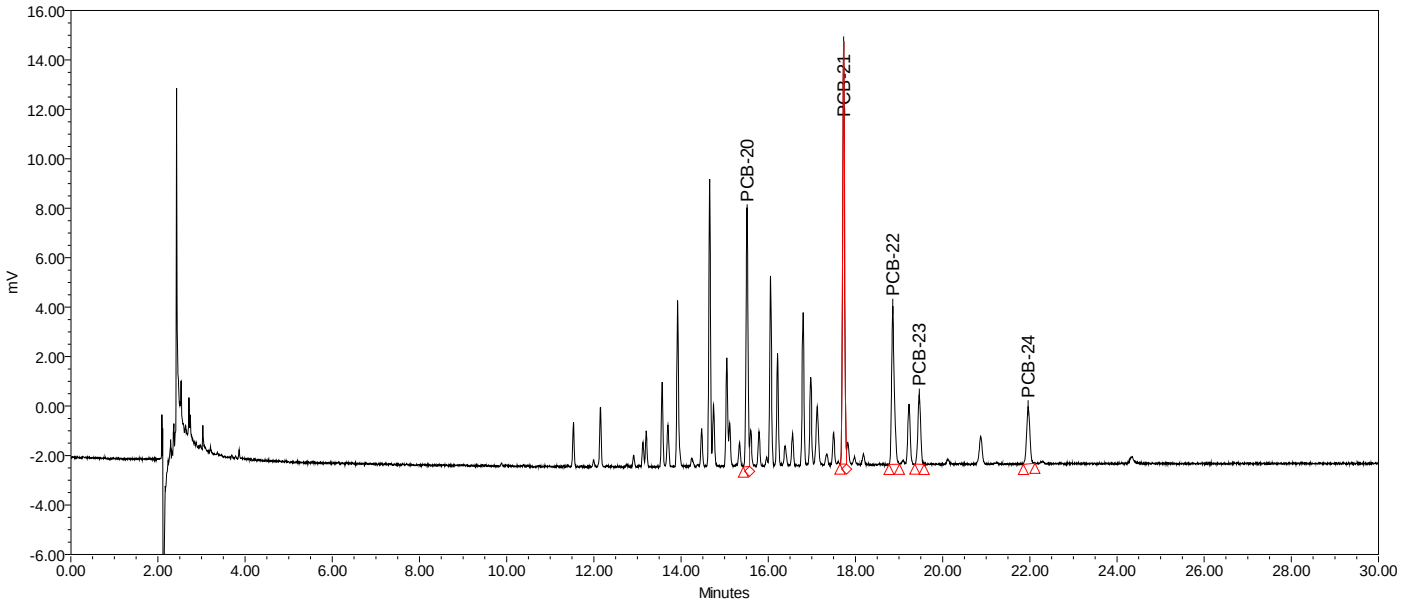


FIGURE 7. A1260 @ 0.500PPM PLOT



ATTACHMENT D: ZB-1 Chromatograms

FIGURE 1. A1016 @ 0.500PPM PLOT

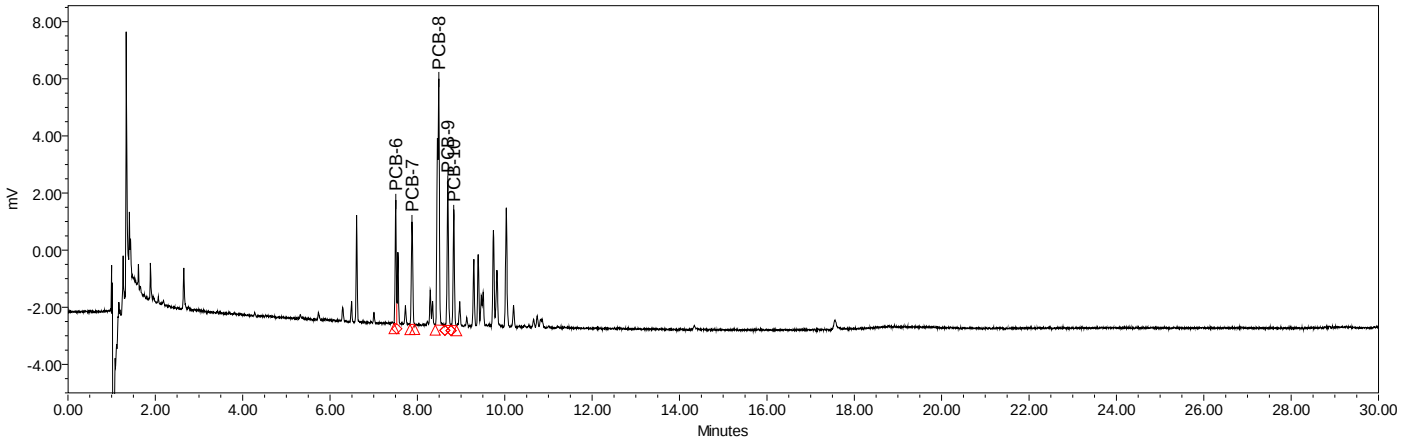


FIGURE 2. A1221 @ 0.500PPM PLOT

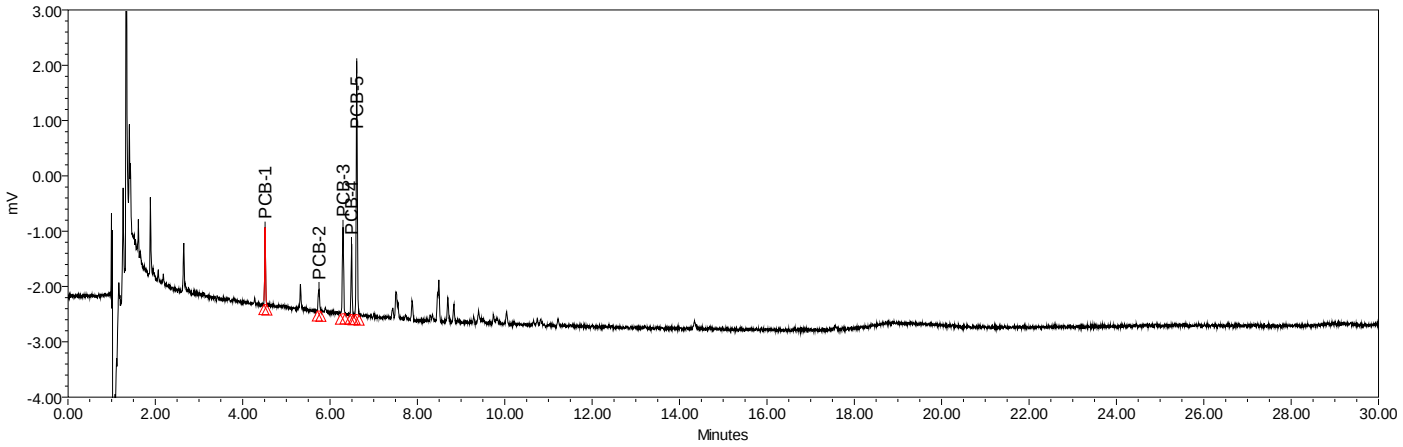


FIGURE 3. A1232 @ 0.500PPM PLOT

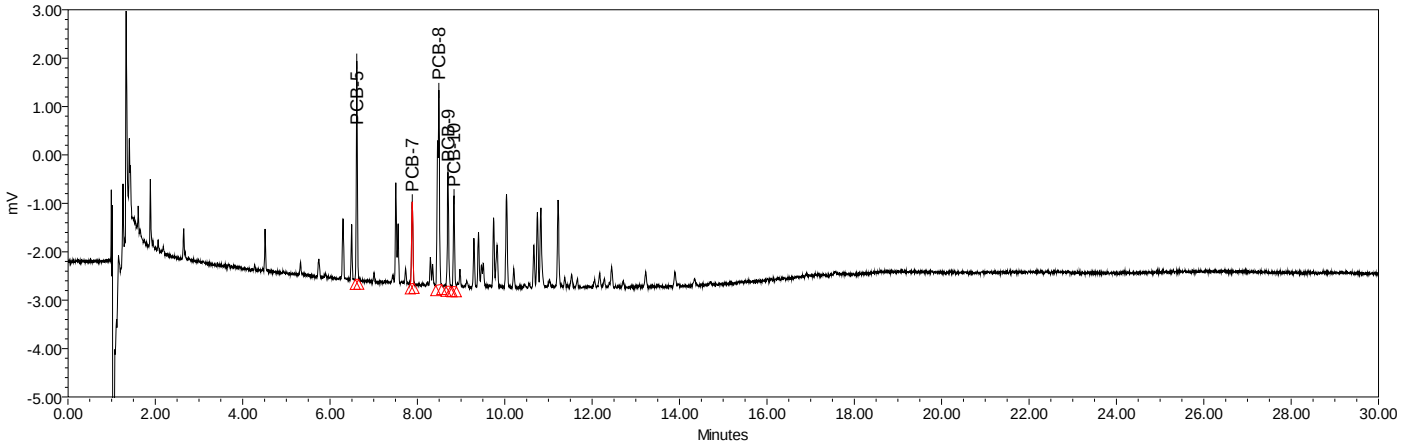


FIGURE 4. A1242 @ 0.500PPM PLOT

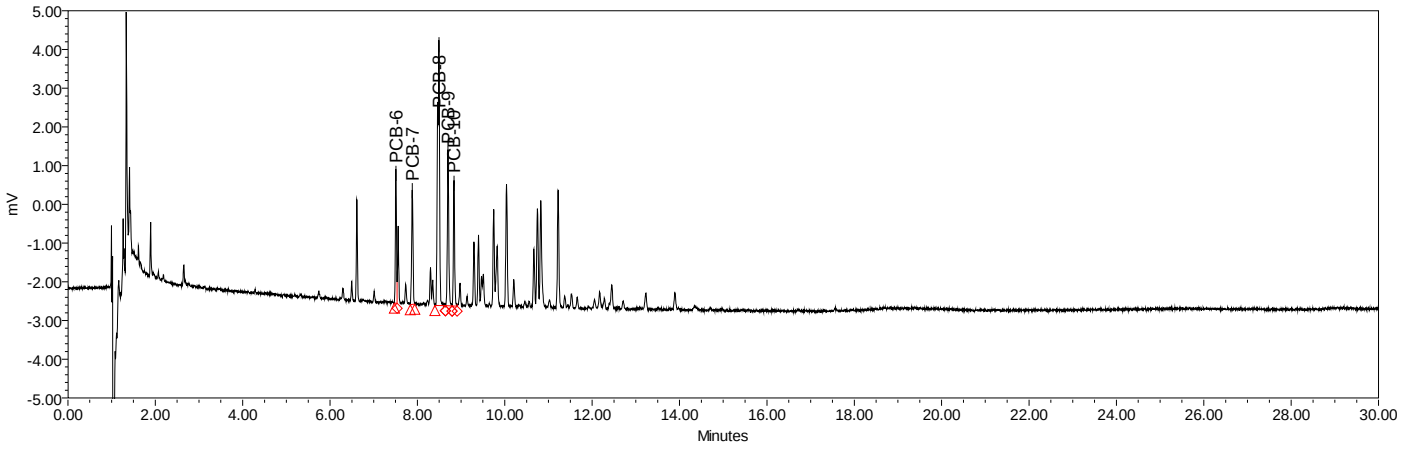


FIGURE 5. A1248 @ 0.500PPM PLOT

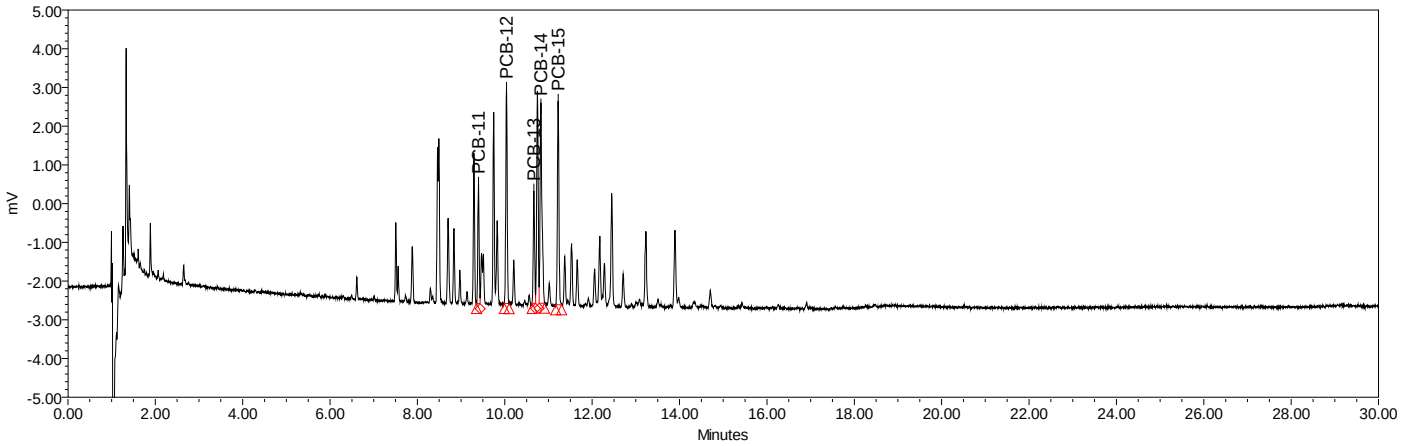


FIGURE 6. A1254 @ 0.500PPM w/ TCMX@10ppb & DCBP@100ppb PLOT

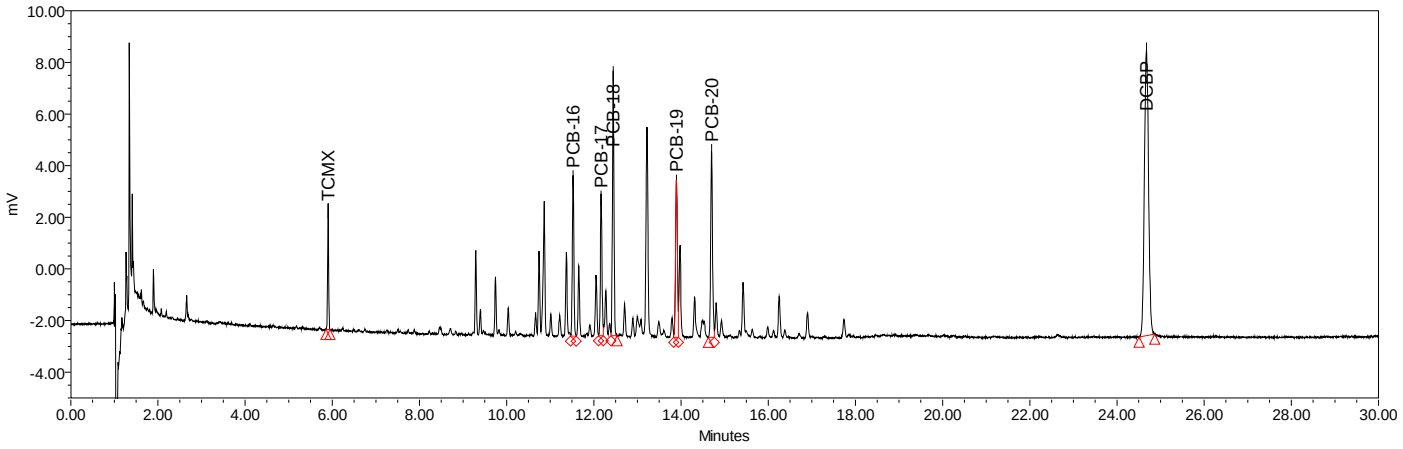
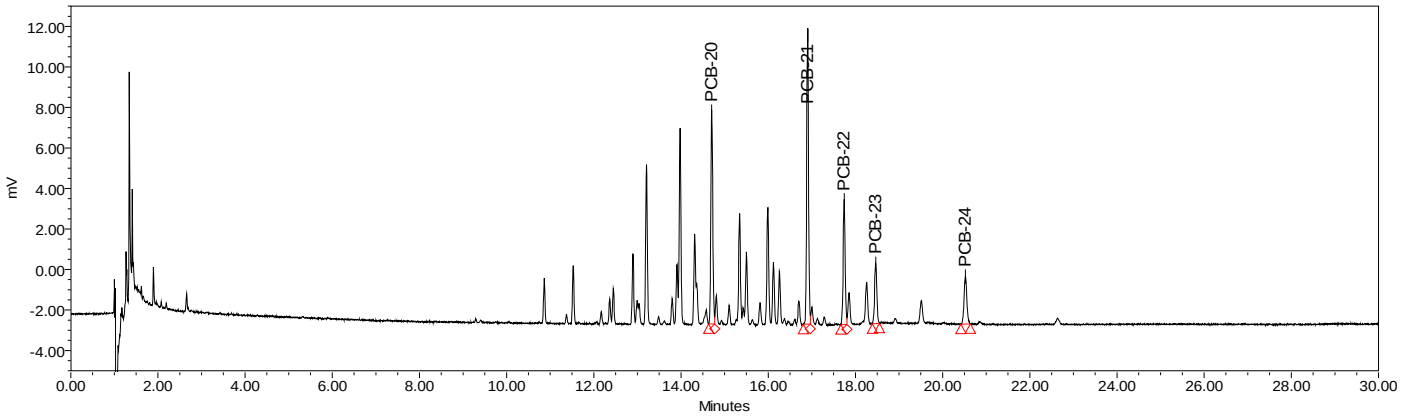


FIGURE 7. A1260 @ 0.500PPM PLOT



Attachment E: Example Calibration Report

Calibration Report: PCB by SW-846 Method 8082
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055

System Name: Instrument_21F **Sample Set Date:** 02/02/2007 12:33:05
Sample Set Name: GC21F_8082CC_020207 **Date Calibrated:** 02/05/2007 10:23:11
User Name: Tom Herold (TomH) **Processing Method:** GC21F_8082_020207
Report Method Name: 8082 CalCurve Summary by RF 01

Calibration Component Summary Table
Component Summary For RF

	Sample Name	A1254-19/20	A1254-20	A1260	A1260-23/24	A1254	A1254-17/18
1	020254A	59.6764	78.3879			106.2382	64.1068
2	020254B	56.2558	74.6686			99.8383	60.5825
3	020254C	56.6040	75.2413			100.2979	60.7165
4	020254D	56.9096	75.6615			100.5600	60.8759
5	020254E	53.3898	71.0511			94.3717	57.0832
Mean		56.567	75.002			100.261	60.673
Std. Dev.		2.235	2.630			4.204	2.486
% RSD		4.0	3.5			4.2	4.1

Calibration Component Summary Table
Component Summary For RF

	Sample Name	TCMX	PCB-16	PCB-17	PCB-18	PCB-19	PCB-20	DCBP
1	020254A	377.950						405.058
2	020254B	382.400						411.024
3	020254C	391.450						425.526
4	020254D	394.860						420.368
5	020254E	383.935						407.430
Mean		386.119						413.881
Std. Dev.		6.895						8.737
% RSD		1.8						2.1

Attachment F: Retention Time Windows: ZB-1 Column

Retention Time Window Study
for Aroclors (PCB) by GC/ECD

Instrument: GC 19F
Column: ZB-1

Analyte	PEAK	Standard 1 500 PPB R.T. Min	Standard 2 50/500 PPB R.T. Min	Standard 3 500 PPB R.T. Min	STD. DEV Min	%RSD	Window +/- Min.
		CS_0327B/A	CS_0328A	CS_0329A			
Aroclor 1016	6	7.927	7.922	7.921	0.0032	0.0406	0.010
	7	8.317	8.312	8.312	0.0029	0.0347	0.009
	8	8.961	8.956	8.956	0.0029	0.0322	0.009
	9	9.178	9.172	9.172	0.0035	0.0378	0.010
	10	9.321	9.317	9.317	0.0023	0.0248	0.007
Aroclor 1221	1	4.894	4.892	4.893	0.0010	0.0204	0.003
	2	6.142	6.142	6.145	0.0017	0.0282	0.005
	3	6.706	6.703	6.705	0.0015	0.0228	0.005
	4	6.904	6.900	6.902	0.0020	0.0290	0.006
	5	7.024	7.021	7.023	0.0015	0.0218	0.005
Aroclor 1232	5	7.025	7.026	7.022	0.0021	0.0296	0.006
	7	8.315	8.318	8.312	0.0030	0.0361	0.009
	8	8.961	8.963	8.957	0.0031	0.0341	0.009
	9	9.178	9.179	9.173	0.0032	0.0350	0.010
	10	9.322	9.324	9.318	0.0031	0.0328	0.009
Aroclor 1242	6	7.925	7.928	7.922	0.0030	0.0379	0.009
	7	8.314	8.318	8.311	0.0035	0.0422	0.011
	8	8.959	8.963	8.956	0.0035	0.0392	0.011
	9	9.176	9.180	9.173	0.0035	0.0383	0.011
	10	9.320	9.324	9.317	0.0035	0.0377	0.011
Aroclor 1248	11	9.906	9.900	9.901	0.0032	0.0325	0.010
	12	10.577	10.573	10.571	0.0031	0.0289	0.009
	13	11.231	11.225	11.225	0.0035	0.0309	0.010
	14	11.396	11.390	11.390	0.0035	0.0304	0.010
	15	11.808	11.803	11.803	0.0029	0.0245	0.009
Arolcor 1254	16	12.121	12.116	12.115	0.0032	0.0265	0.010
	17	12.793	12.786	12.785	0.0044	0.0341	0.013
	18	13.075	13.069	13.070	0.0032	0.0246	0.010
	19	14.572	14.567	14.566	0.0032	0.0221	0.010
	20	15.394	15.389	15.389	0.0029	0.0188	0.009
Arolcor 1260	20	15.396	15.398	15.392	0.0031	0.0198	0.009
	21	17.668	17.670	17.664	0.0031	0.0173	0.009
	22	18.611	18.613	18.606	0.0036	0.0194	0.011
	23	19.417	19.421	19.415	0.0031	0.0157	0.009
	24	21.729	21.731	21.722	0.0047	0.0218	0.014
TCMX (SURROGATE)	Surr.	6.311	6.307	6.307	0.0023	0.0366	0.007
DCB (SURROGATE)	Surr.	26.432	26.421	26.421	0.0064	0.0240	0.019

Attachment G: Retention Time Windows ZB-5 Column

Retention Time Window Study
for Aroclors (PCB) by GC/ECD

Instrument: GC 19B
Column: ZB-5

Analyte	PEAK	Standard 1 500 PPB R.T. Min	Standard 2 500 PPB R.T. Min	Standard 3 500 PPB R.T. Min	STD. DEV Min	%RSD	Window +/- Min.
		CS_0327B/A	CS_0328A	CS_0329A			
Aroclor 1016	6	8.260	8.256	8.256	0.0023	0.0280	0.007
	7	8.669	8.665	8.665	0.0023	0.0266	0.007
	8	9.277	9.272	9.272	0.0029	0.0311	0.009
	9	9.502	9.498	9.498	0.0023	0.0243	0.007
	10	9.685	9.680	9.679	0.0032	0.0332	0.010
Aroclor 1221	1	5.480	5.479	5.476	0.0021	0.0380	0.006
	2	6.618	6.615	6.611	0.0035	0.0531	0.011
	3	7.086	7.084	7.084	0.0012	0.0163	0.003
	4	7.291	7.289	7.288	0.0015	0.0210	0.005
	5	7.410	7.406	7.407	0.0021	0.0281	0.006
Aroclor 1232	5	7.408	7.412	7.409	0.0021	0.0281	0.006
	7	8.667	8.671	8.667	0.0023	0.0266	0.007
	8	9.278	9.278	9.274	0.0023	0.0249	0.007
	9	9.502	9.505	9.501	0.0021	0.0219	0.006
	10	9.684	9.686	9.681	0.0025	0.0260	0.008
Aroclor 1242	6	8.258	8.261	8.255	0.0030	0.0363	0.009
	7	8.667	8.670	8.664	0.0030	0.0346	0.009
	8	9.275	9.279	9.272	0.0035	0.0379	0.011
	9	9.501	9.506	9.498	0.0040	0.0425	0.012
	10	9.682	9.686	9.679	0.0035	0.0363	0.011
Aroclor 1248	11	10.202	10.196	10.198	0.0031	0.0300	0.009
	12	10.936	10.930	10.932	0.0031	0.0279	0.009
	13	11.545	11.540	11.541	0.0026	0.0229	0.008
	14	11.748	11.743	11.742	0.0032	0.0274	0.010
	15	12.219	12.215	12.217	0.0020	0.0164	0.006
Arolcor 1254	16	12.402	12.397	12.397	0.0029	0.0233	0.009
	17	13.175	13.170	13.168	0.0036	0.0274	0.011
	18	13.469	13.463	13.461	0.0042	0.0309	0.012
	19	14.942	14.935	14.934	0.0044	0.0292	0.013
	20	15.812	15.805	15.805	0.0040	0.0256	0.012
Arolcor 1260	20	15.808	15.810	15.804	0.0031	0.0193	0.009
	21	18.082	18.085	18.079	0.0030	0.0166	0.009
	22	19.272	19.274	19.268	0.0031	0.0159	0.009
	23	19.911	19.910	19.912	0.0010	0.0050	0.003
	24	22.558	22.557	22.552	0.0032	0.0143	0.010
TCMX (SURROGATE)	Surr.	6.658	6.653	6.653	0.0029	0.0434	0.009
DCB (SURROGATE)	Surr.	27.519	27.509	27.498	0.0105	0.0382	0.032

Attachment H:

Table 1. Example of MDL Study ZB-1 Column

Compound:	A1221	Analysis:	EPA METHOD 8082
Matrix:	SOIL/SOLID	Instrument:	GC-18F/ LEAP
Extraction:	ASE	Column:	ZB-1
Spike conc:	75.0 ug/kg		

NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ug/kg	Percent Recovery (%)
AJ00194	01/17/06	1221/1254 #1 ASE	05/04/06	77.5	103%
AJ00195	01/17/06	1221/1254 #2 ASE	05/04/06	68.6	91%
AJ00196	01/17/06	1221/1254 #3 ASE	05/04/06	69.2	92%
AJ00197	01/17/06	1221/1254 #4 ASE	05/04/06	74.4	99%
AJ00198	01/17/06	1221/1254 #5 ASE	05/04/06	74.5	99%
AJ00199	01/17/06	1221/1254 #6 ASE	05/04/06	72.0	96%
AJ00200	01/17/06	1221/1254 #7 ASE	05/04/06	77.1	103%
AJ00201	01/17/06	1221/1254 #8 ASE	05/04/06	74.5	99%

		Number (n):	8
One sided Student's t values (t)		AVG:	73.5 ug/kg
at the 99% confidence level.		STD (s):	3.31 ug/kg
Number (n)	(t) value	%RSD:	4.50%
7	3.143	MDL:	9.91 ug/kg
8	2.998	PQL:	49.6 ug/kg
		VALID:	valid

Table 2. Example of MDL Study ASE Extraction ZB-5 Column

Compound:	A1221	Analysis:	EPA METHOD 8082
Matrix:	SOIL/SOLID	Instrument:	GC-18B/LEAP
Extraction:	ASE	Column:	ZB-5
Spike conc:	75.0 ug/kg		

NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ug/kg	Percent Recovery (%)
AJ00194	01/17/06	1221/1254 #1 ASE	07/04/06	81.9	109%
AJ00195	01/17/06	1221/1254 #2 ASE	07/04/06	73.2	97.6%
AJ00196	01/17/06	1221/1254 #3 ASE	07/04/06	79.4	105.9%
AJ00197	01/17/06	1221/1254 #4 ASE	07/04/06	74.2	98.9%
AJ00198	01/17/06	1221/1254 #5 ASE	07/04/06	75.0	100.1%
AJ00199	01/17/06	1221/1254 #6 ASE	07/04/06	82.0	109.3%
AJ00200	01/17/06	1221/1254 #7 ASE	07/04/06	77.9	104%
AJ00201	01/17/06	1221/1254 #8 ASE	07/04/06	81.1	108.1%

		Number (n):	8
One sided Student's t values (t) at the 99% confidence level.		AVG:	78.1 ug/kg
		STD (s):	3.56 ug/kg
Number (n)	(t) value	%RSD:	4.56%
7	3.143	MDL:	10.69 ug/kg
8	2.998	PQL:	53.4 ug/kg
		VALID:	valid

Table 3. Example of MDL Study SOX Extraction ZB-1 Column

Compound:	A1221	Analysis:	EPA METHOD 8082
Matrix:	SOIL/SOLID	Instrument:	GC-18F/LEAP GC PAL
Extraction:	SOX	Column:	ZB-1
Spike conc:	75.00 ug/kg		

NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ug/kg	Percent Recovery (%)
AJ00234	01/17/06	1221/1254 #1 SOX	04/19/06	81.0	108.0%
AJ00235	01/17/06	1221/1254 #2 SOX	04/19/06	79.4	106%
AJ00236	01/17/06	1221/1254 #3 SOX	04/19/06	77.0	102.7%
AJ00237	01/17/06	1221/1254 #4 SOX	04/19/06	81.1	108.1%
AJ00238	01/17/06	1221/1254 #5 SOX	04/19/06	77.1	102.8%
AJ00239	01/17/06	1221/1254 #6 SOX	04/19/06	73.7	98%
AJ00240	01/17/06	1221/1254 #7 SOX	04/19/06	71.8	95.7%
AJ00241	01/17/06	1221/1254 #8 SOX	04/19/06	77.4	103%

		Number (n):	8	
One sided Student's t values (t)		AVG:	77.31	ug/kg
at the 99% confidence level.		STD (s):	3.293	ug/kg
Number (n)	(t) value	%RSD:	4.26%	
7	3.143	MDL:	9.871	ug/kg
8	2.998	PQL:	49.36	ug/kg
		VALID:	valid	

Table 4. Example of MDL Study SOX Extraction ZB-5 Column

Compound:	A1221	Analysis:	EPA METHOD 8082
Matrix:	SOIL/SOLID	Instrument:	GC-18B/LEAP GC PAL
Extraction:	SOX	Column:	ZB-5
Spike conc:	75.00 ug/kg		

NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ug/kg	Percent Recovery (%)
AJ00234	01/17/06	1221/1254 #1 SOX	07/01/06	79.2	105.7%
AJ00235	01/17/06	1221/1254 #2 SOX	07/01/06	74.7	100%
AJ00236	01/17/06	1221/1254 #3 SOX	07/01/06	80.2	107.0%
AJ00237	01/17/06	1221/1254 #4 SOX	07/01/06	79.8	106.3%
AJ00238	01/17/06	1221/1254 #5 SOX	07/01/06	81.9	109.2%
AJ00239	01/17/06	1221/1254 #6 SOX	07/01/06	78.3	104%
AJ00240	01/17/06	1221/1254 #7 SOX	07/01/06	85.2	113.6%
AJ00241	01/17/06	1221/1254 #8 SOX	07/01/06	82.6	110%

		Number (n):	8	
One sided Student's t values (t)		AVG:	80.23	ug/kg
at the 99% confidence level.		STD (s):	3.146	ug/kg
Number (n)	(t) value	%RSD:	3.92%	
7	3.143	MDL:	9.432	ug/kg
8	2.998	PQL:	47.16	ug/kg
		VALID:	valid	

Table 5. Example of MDL Study PUF Extraction ZB-1 Column

Compound:	A1221	Analysis:	EPA METHOD 8082
Matrix:	PUF	Instrument:	GC-19F/8400
Extraction:	SOX	Column:	ZB-1
Spike conc:	0.150 ug		

NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ug	Percent Recovery (%)
AJ00508	02/08/06	TO-10A 1221/1254 #1	02/10/06	0.138	91.8%
AJ00509	02/08/06	TO-10A 1221/1254 #2	02/10/06	0.130	86.8%
AJ00510	02/08/06	TO-10A 1221/1254 #3	02/10/06	0.141	93.7%
AJ00511	02/08/06	TO-10A 1221/1254 #4	02/10/06	0.150	99.8%
AJ00512	02/08/06	TO-10A 1221/1254 #5	02/10/06	0.135	90.2%
AJ00513	02/08/06	TO-10A 1221/1254 #6	02/10/06	0.142	94.6%
AJ00514	02/08/06	TO-10A 1221/1254 #7	02/10/06	0.140	93.1%
AJ00515	02/08/06	TO-10A 1221/1254 #8	02/10/06	0.134	89.2%

		Number (n):	8
One sided Student's t values (t)		AVG:	0.139 ug
at the 99% confidence level.		STD (s):	0.006 ug
Number (n)	(t) value	%RSD:	4.25%
7	3.143	MDL:	0.018 ug
8	2.998	PQL:	0.088 ug
		VALID:	valid

Table 6. Example of MDL Study PUF Extraction ZB-5 Column

Compound:	A1221	Analysis:	EPA METHOD 8082
Matrix:	PUF	Instrument:	GC-19B/8400
Extraction:	SOX	Column:	ZB-5
Spike conc:	0.150 ug		

NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ug	Percent Recovery (%)
AJ00508	02/08/06	TO-10A 1221/1254 #1	02/10/06	0.143	95.3%
AJ00509	02/08/06	TO-10A 1221/1254 #2	02/10/06	0.127	84.8%
AJ00510	02/08/06	TO-10A 1221/1254 #3	02/10/06	0.135	90.2%
AJ00511	02/08/06	TO-10A 1221/1254 #4	02/10/06	0.134	89.0%
AJ00512	02/08/06	TO-10A 1221/1254 #5	02/10/06	0.134	89.1%
AJ00513	02/08/06	TO-10A 1221/1254 #6	02/10/06	0.136	90.4%
AJ00514	02/08/06	TO-10A 1221/1254 #7	02/10/06	0.143	95.2%
AJ00515	02/08/06	TO-10A 1221/1254 #8	02/10/06	0.140	93.3%

One sided Student's t values (t) at the 99% confidence level.	
Number (n)	(t) value
7	3.143
8	2.998

Number (n):	8	
AVG:	0.136	ug
STD (s):	0.005	ug
%RSD:	3.91%	
MDL:	0.016	ug
PQL:	0.080	ug
VALID:	valid	

Notes

STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

NE158_05.DOC
REVISION NUMBER: 05

STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF LIPIDS FROM FISH AND BIOTA MATERIAL

COPY #___

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STANDARD OPERATING PROCEDURE

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1.0 TITLE/PURPOSE

Title:

Standard Operating Procedure for the extraction and analysis of % lipids from fish and biota material.

Purpose:

This SOP provides the method for the extraction of lipids from fish tissue.

2.0 APPLICABLE MATRICES

This test method is appropriate for fish and biota tissue. This method may be restricted to use by or under the supervision of a chemist knowledgeable in the area of sample extraction and clean-up. The chemist should further be aware of the proper care and handling of Polychlorinated Biphenyls (PCBs) as well. The chemist must have an understanding of the methods and requirements of USEPA-SW-846A "Test Methods for Solid Wastes" Volume 1B: Lab Manual, 3rd edition. Methods 3540, 3500, 2500A. The chemist must also be certified to perform the procedure by an approved instructor.

3.0 DETECTION LIMIT

The detection limit is defined by the precision of the balance used while weighting out the sample. The detection limit for the Mettler Toledo PL303 balance is 0.001g. If there is a limited amount of oil to detect it may be necessary to utilize the Mettler Toledo AG204

balance with a detection limit of 0.0001g.

4.0 SCOPE AND APPLICATION

The following procedure is used by NEA for the extraction of fish tissue for lipid analysis, though it may in part be adapted for other biota extractions.

5.0 SUMMARY OF TEST METHOD

The extraction of lipids from fish and biota material: biota samples are extracted via soxhlet extraction. Then the samples are solvent exchanged into pure hexane and set to 25ml using a 25ml volumetric. Once set to volume 10ml are taken and placed in a properly labeled and pre-weighed tin. The extracted solvent is then blown dry in the tin utilizing a micro blowdown apparatus. Using the initial weight and the final weight of the tin the % lipid concentration in the sample material can be calculated (see calculations).

6.0 DEFINITIONS

Blank: A non site sample created in the laboratory to evaluate cross contamination of the sample set

Lab Control Standard: A non site sample created in the laboratory to which a known amount of target analyte is added for assessment of analyte recovery efficiency

Lab Control Standard Duplicate: An exact copy of the Lab Control Standard to further assess analyte recovery efficiency

Matrix Spike: A site sample to which a known amount of target analyte is added for assessment of analyte recovery efficiency.

Matrix Spike Duplicate: An exact copy of the Matrix Spike utilizing the same site sample and known amount of target analyte for assessment of analyte recovery efficiency

Relative Percent Difference (RPD): a quality control measure designed to quantitate the difference in percentages for lipids/total solids, etc. of two identical samples.

7.0 INTERFERENCES:

Laboratory technicians should exercise caution when completing % lipid analysis. Technician should be sure that no foreign material is included with extract when transferred to aluminum boat. For example, glass particles from pipettes may cause a false result and compromise the integrity of the % lipid analysis.

8.0 SAFETY

Polychlorinated biphenyls should be treated with extreme caution; as a class of chemical compounds they possess both toxic and carcinogenic properties. The chemist should have received in-house safety training and should know the location of first aid equipment and the emergency spill/clean-up equipment, before handling any apparatus or equipment. Safety glasses and gloves must be worn when handling glassware and samples.

9.0 EQUIPMENT AND SUPPLIES

9.1 Water Cooled Condenser: Pyrex 45/50 #3840-MCO.
9.1 250mL Round Bottom Flask: Pyrex #4100
9.1 Soxhlet Repetitive Flushing (reflux) Unit: 45/50 Pyrex #3740-M.
9.1 Heating Mantle: Type "VF" laboratory heating mantle #HM0250VF1. (Or equivalent)
9.1 Heating Mantle Controller: Glass-Col #PL3122 Minitwin (or equivalent) regulates temperature control of the mantle.
9.1 Analytical Balance: Mettler PL-303 used to determine sample mass.
9.1 Chiller: Pump driven water circulating cooling system cool flow #75 NESLABS Instruments, Inc. (or equivalent)
9.1 Turbo Vap Evaporator: Zymark #ZW640-3.
9.1 Turbo Vap Evaporator concentrator tubes: Zymark 250mL, 0.5mL endpoint.
9.1 Beakers: Assorted Pyrex: 250mL, 600mL, and 1000mL, used for liquid containment and pipet storage.
9.1 Vials: glass, 40mL vial & 4-dram (with polyseal sealed cap) (20 mL & 10 mL) capacity, for sample extracts.
9.1 Vial Rack: Plastic rack used to hold vials, during all phases of the extract processing.
9.1 Pipettes: S/P Disposable Serological Borosilicate Pipettes.
   1. 1mL X 1/10
   2. 5mL X 1/10
   3. 10mL X 1/10
   4. Fisher Pasteur Borosilicate glass pipet 9" (or equivalent)
9.1 Beakers: Assorted Pyrex: 250mL, 600mL, and 1000mL.
9.1 Weighing Boats: Tin weighing boats for percent lipid weight determination.
9.1 Metal spatula.
9.1 4 oz Jars: Industrial Glassware

10.0 REAGENTS AND STANDARDS

10.1 Cellulose Extraction Thimble: Contains sample during soxhlet extraction.
10.2 Sodium Sulfate: J.T. Baker #3375-05 Anhydrous, Granular (12-60 Mesh). Used for the laboratory method blank and laboratory control spike.
10.3 Boiling Chips: Chemware PTFE Boiling Stones P#0919120 (or equivalent)
10.4 Hexane: High Purity Solvent Baxter (Burdick/Jackson) #UN1208. (Or equivalent)
10.5 Acetone: High Purity Solvent Baxter (Burdick/Jackson) #UN1090. (Or equivalent)
10.6 1:1 Hexane/Acetone: 50%/50% by volume solvent mixture prepared in the lab.

16.7 Magnesium

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT and STORAGE

All samples should remain frozen at all times unless being tested. Fish usually arrive whole bodied or already filleted. Once received the sample must be ground and homogenized so that it may be analyzed. The homogenized fish tissue should be held for 6 to 12 months. The fish solvent extracts should be held for 3 months. Some clients may request that the body and/or head of fish be saved once the fillets are cut out. Other biota material may have other specifications stated specifically for that project.

12.0 QUALITY CONTROL

12.1 The method blank is used as a measure to ensure that only the lipid content in the sample is responsible for the weight change allowing for the calculation of % lipids in the sample.

12.2 The Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are further controls which test the method to ensure proper lipid recovery is performed by calculating their RPD (see section 18.0).

13.0 CALIBRATION AND STANDARDIZATION

Both the PL303 and the AG201 analytical balances should be calibrated daily to ensure proper measurements are being made.

14.0 PROCEDURES

14.1 Sample Preparation

14.1.1 Throughout the entire process it should be noted that if the chemist encounters any problems or difficulties with any samples or steps involved, all work should STOP! Any problems should be brought to the attention of the supervisor and documented in the extraction logbook.

14.1.2 Before any steps are taken, the chemist should first review the sample job folder and fill in the appropriate spaces on the internal sample tracking form and initial.

14.1.3 The fish samples are usually received as fillets and must be processed to produce a homogenous material prior to extraction. Once the sample has been processed, the sample is cut up into small enough pieces to fit into a standard manual meat grinder. An acetone rinsed Pyrex tray should be placed under the grinder exit to catch the minced tissue. After the fish has been ground once, it should be ground again to ensure complete homogenization of the sample. The ground fish is then placed into an appropriate-sized jar and labeled. The sample is then placed in the freezer for storage until the extraction process is begun.

14.2 Procedure: Sample Extraction

14.2.3 Rinse the thimbles with hexane; allow them to dry out in a 4 ounce jar in a fume hood.

14.2.4 Fill a Pyrex pan with ice cubes and cold water about 1/2 inch deep. As the samples are weighed out, place the jars in the Pyrex pan to chill for at least 15 minutes prior to the drying step.

14.2.5 Into a tarred a 4oz. jar accurately weigh to the nearest 0.001g using an analytical balance about 10 grams of tissue. Record this weight in the laboratory extraction logbook and LIMS system. Place the jar containing the sample into the Pyrex pan to chill. Repeat for remaining samples.

NOTE: ALL SAMPLE CONTAINERS ARE TO BE RETURNED TO THE APPROPRIATE REFRIDGERATOR. FOR ALL EMPTY SAMPLES CONTAINERS, SEE THE CHEMICAL HYGIENE PLAN FOR PROPER DISPOSAL.

14.2.6 After the sample has been sufficiently chilled, add approximately 10 g. of a 1:1 mix of magnesium sulfate/sodium sulfate to the sample and mix well with a clean metal spatula. If the sample has not dried after a few minutes, another 10 g. may be added. Once the sample is well dried and free flowing, transfer the sample to an extraction thimble. Repeat with remaining samples. Set empty mixing jar in glass disposal bin and stirring utensil aside for cleaning. **Be careful not to add too much drying agent to the sample; if too much is added, the sample may not fit completely in the thimble. In this case the sample will have to be split into two different soxhlets.**

14.2.7 Add 200 mL of a 1:1 mixture of hexane/acetone to a 250 mL round bottom flask. Add several boiling chips. Place a soxhlet extractor on top of the round bottom flask. Label the round bottom with a sample number and use a pair of long-handled tweezers to place the corresponding thimble into the soxhlet extractor. After all samples have been processed add the specified surrogate and matrix spikes required directly into thimble.

14.2.8 Rinse the inside and the outside connecting joints of the condenser units that will be used to condense the extraction solvent during the soxhlet extraction of the sample. Turn on chiller to cool the condensers.

14.2.9 Place the round bottom flask with attached soxhlet extractor onto a heating mantle and attach condenser unit. Turn corresponding thermostats on to setting 5. Double check soxhlets at this time for any cracks or chips which may leak solvent. Once the solvent begins to boil, a flushing action of once every two to three minutes should be achieved.

18 ± 2

14.2.10 The samples should be extracted overnight for a ~~minimum of 16 h~~ours. Once the sample has finished extracting (usually in the morning), turn the heating mantle off and allow samples to cool to room temperature. Once cool, rinse the inside of the condenser with several pipette volumes of hexane. Disengage the soxhlet and condenser unit and rinse the joint off as well into the soxhlet.

14.2.11 Move soxhlet units into a chemical fume hood and flush the remaining solvent from

the soxhlet extractor by tipping the soxhlet. Using a pair of long-handled tweezers, pull the thimbles out of the soxhlets one at a time and allow them to drip dry by balancing the thimbles on the tops of the soxhlets. Once dry, remove the thimbles to a sheet of aluminum foil for total solvent evaporation and disposal.

14.2.12 Rinse the soxhlet with several pipetfuls of hexane and tip again to drain into the round bottom. Set aside the soxhlet at this time. Procure the same number of turbo tubes as there are samples. Using an individual turbotube stand, label a turbotube with the corresponding sample ID number and place in the holder. Pour the contents of the round bottom into the turbotube, using a pipette and hexane to rinse the last drop out of the mouth of the round bottom. Rinse the round bottom with several pipetfuls of hexane, swirl gently, and decant into same turbo tube. Repeat this step twice for same sample, and then repeat all preceding steps for all other samples.

14.2.13 All glassware must be rinsed with technical grade (tech)-acetone or a "for rinsing-only" labeled solvent, and dried in the hood before other cleaning steps.

14.3 **Solvent Reduction: TurboVap Evaporator System**

14.3.3 The Turbovap evaporator system is used in place of the Kuderna Danish (KD) - concentrator apparatus.

The turbovap uses a heated water bath and positive pressure nitrogen flow/vortex action. The unit maintains a slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract, which allows fractional reduction of the solvents without loss of higher boiling point analytes.

14.3.4 Turn the unit on (switch is located on the backside of the unit) and allow to heat up to the specified temperature for individual solvent use. This is indicated by the "Heating" display light, located above the temperate control knob on the right side of the unit. The system is at the proper temperature when the "At Temperature" light is lit. This is located above the "Heating" display light. The temperature of the unit should be set at 40 ± 2°C.

14.3.5 As a precaution the TurboVap system regulators should be checked to assure that no residual gas pressure remains within the system and that the gas cylinder valve and gas pressure regulators are both off before placing samples in the apparatus. Residual gas pressure may cause splashing and cross contamination of samples. To bleed the system of residual gas pressure place an empty turbo tube into the water bath and close the lid. Make sure that the nitrogen gas cylinder valve is turned off and slowly turn on the gas pressure regulator. Bleed any residual gas until the regulator output pressure gauge reads "0" psi. Proceed to 8.3.4.

14.3.6 Place the turbo tube containing the samples into the Turbovap and close the lid. Turn on the gas cylinder valve first and then begin slowly turning the pressure regulator on. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the reduction.

14.3.7 The process for solvent (hexane/acetone) reduction takes approximately 20-30 minutes. Do not leave the unit unattended as extracts may be blown to dryness.

14.3.8 Concentrate the solvent to approximately 10.0 mL. Remove the samples from the turbovap and place in the rack. The remaining solvent will consist largely of hexane since the acetone component is fractionally removed at a faster rate than hexane; however, a solvent exchange with hexane should be completed by filling the turbo tube up with approximately 50mls of hexane and concentrating back down to 10.0mls to ensure the acetone has been entirely removed.

NOTE: Not all samples will evaporate at the same rate; sample extracts containing large amounts of lipids or other non-volatile liquids may stop reducing before the 10.0 mL point is achieved. Samples that stop reducing should be removed as soon as possible.

14.3.9 Quantitatively transfer the sample extract with a disposable transfer pipette into an appropriate volumetric flask (25mL for biota extracts) with three 2mL hexane rinses. After the sample has been transferred, rinse the disposable transfer pipet with 0.5mL of hexane into the volumetric flask. Add hexane to the volumetric meniscus mark. Invert the volumetric flask at least three times to mix completely. Decant the contents into a pre-labeled 40mL vial.

14.3.10 All dirty glassware must be rinsed with tech-acetone or a "For Rinsing-Only" labeled solvent and dried in the fume hood before being washed.

14.4 **Sample Extract concentration**

NOTE: NO CLEAN-UP STEPS ARE PERFORMED FOR PERCENT LIPID DETERMINATION.

14.4.1 Weigh a pre-labeled tin weighing boat for each sample extract. Record the weight in the percent lipid log book and LIMS system.

14.4.2 Shake samples for a few seconds then using a 10mL pipet, transfer 10mL of the extract into the corresponding weighing boat.

14.4.3 Concentrate the extract in the weighing boat using the nitrogen micro-blowdown. Place a maximum of 3 weighing boats at one time on the nitrogen blow down. Set the temperature at 40C and the pressure at 40 PSI.

14.4.4 After concentration has been completed, place the weighing boats into the desiccators for at least 4 hours.

14.4.5 Weigh sample weighing boats and record the weight in the percent lipid logbook and LIMS system. Using the below equation calculate percent lipids.

15.0 CALCULATIONS

15.1 Calculate percent lipids as follows:

$$\text{PERCENT LIPIDS} = \frac{Vf - Ve \times 2.5 \times 100}{\text{Sample weight (g)}}$$

Vf = weight of tin tray with sample (g)
Ve = weight of empty tin tray (g)

15.2 $$PQL = \frac{0.001(\text{or } 0.0001)^*}{\text{sample weight}} \times 100$$

*Depending on which balance was used to weigh the sample. The PL303 analytical balance would use 0.001 while the AG204 would use 0.0001.

16.0 METHOD PERFORMANCE

Method Performance is measured through the use of laboratory duplicates (Matrix Spike/Matrix Spike Duplicates). The Laboratory Information Management System (LIMS) maintains records of laboratory duplicate analysis.

17.0 POLLUTION PREVENTION

See NEA168.SOP

18.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

18.1 Laboratory Duplicate % RPD $\leq$ 20
18.2 Method Blank % lipid $\leq$ PQL

19.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

19.1 Initially attempt to re-weight the sample making sure the balance is properly calibrated.
19.2 If data is still out of control then it is necessary to re-homogenize the sample and then re-extract and re-calculate.

20.0 CONTINGINCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

After all corrective actions are taken with no change or there is not enough sample material to perform corrective actions, report to client with what data is available detailing how it is out of control.

21.0 WASTE MANAGEMENT

See NEA054.SOP, NEA083.SOP, and NEA089.SOP

22.0 REFERENCES

10.1 SW-846 methods 3500A & 3600A; United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Vol.1B, Cincinnati, OH 45268.

10.2 Guide to Environmental Analytical Methods, Genium Publishing Corporation, Schenectady, NY 12304

23.0 TABLES, DIAGRAMS, FLOWCHARTS and VALIDATION DATA

None

STANDARD OPERATING PROCEDURE FOR GEL-PERMEATION CLEANUP (GPC) SW846 METHOD 3640

NE226_01.SOP
Revision 01

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1.0 Identification of Test Method

- This method is used as a clean-up procedure to remove lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids, and dispersed high-molecular weight compounds from sample extracts to be analyzed via GC or GC/MS.

- GPC is appropriate for both polar and non-polar analytes, therefore, it can be effectively used to cleanup extracts containing a broad range of analytes.

2.0 Applicable Matrix

- This method is generally used with biota/tissue samples but is applicable to other solids or liquids as well.

3.0 Detection Limit

- N/A

4.0 Scope and Application, Including Components to be Analyzed

- This method provides guidance for the cleanup of sample extracts for semi-volatiles, pesticides and polychlorinated biphenyls (PCBs).

- Other analytes may also be appropriate for this cleanup technique, however, recovery through the GPC should be >70%.

5.0 Summary of Test Method

- The column is packed with the required amount of preswelled packing gel and flushed with solvent for an extended period. The column is calibrated for appropriate fraction times, and checked for QC. The sample extract is loaded into the column, collected, and then concentrated.

6.0 Definitions

- Quality Control (QC): A set of measures within sample analysis methodology to insure the process is in control.

7.0 Interferences

- To insure quality analysis, precautions must be taken to avoid contamination.
  - Glassware must be scrupulously cleaned.
  - High purity solvents must be used.
  - Reagent reservoirs must be covered at all times.

8.0 Safety

- Dichloromethane is the principal solvent used for this method. Dichloromethane is a suspected carcinogen. When working with this solvent a maximum effort should be made to properly utilize fume hoods.

- Calibration standards contain possible carcinogens. Prepare all standards, do all spiking and make all dilutions in a fume hood.

- Safety glasses and gloves are required. A lab coat or apron is strongly recommended.

- Before beginning work, always consult the chemical hygiene plan or ask the chemical hygiene officer if you need information about how to work with certain chemicals.

9.0 Equipment and Supplies

- J2 Scientific Accuprep™ GPC chromatography system.

- UV Detector – D-Star Fixed Wavelength detector model # DFW-20.

- GPC Columns: Traditional S-X3 (pesticides & PCBs), J2 Scientific (CO100), High Pressure (SVOC), Waters Envirogel 19x300mm (WAT36554), Waters Envirogel 19x150mm (WAT36555) guard column.

- Software: J2 Scientific, Accuprep™ Chromatography Application software version 11.8, AccuChrome™ GPC cleanup Application software version 2.1.0.9.

- Auto sampler Vials – 10mL, J2 Scientific (BV16100T-CS).

- Vial Caps – J2 Scientific 16mm (BV106).

- Cap Liners – J2 Scientific PTFE Disc, 13mm, (BV016T-CS).

- Syringes – Norm-Ject 10mL single use, VWR (53548-018), or equivalent.

- Syringe Filters – Acrodisc Premium 25mm, 0.45um PTFE membrane, VWR (28143-866), or equivalent.

10.0 Reagents and Standards

- Dichloromethane: High purity solvent, Burdick & Jackson.

- Toluene: High purity solvent, Burdick & Jackson.

- GPC Calibration Standard Solution: 0.2-250 mg/mL, Ultra Scientific (CLP-340).

Analyte	Concentration
corn oil	250 mg/mL
bis(2-ethylhexyl)phthalate	10 mg/mL
methoxychlor	2 mg/mL
perylene	0.2 mg/mL
sulfur	0.8 mg/mL

  - GPC Calibration Standard @ 2-2500µg/mL: To a 10mL volumetric flask containing 5mL of dichloromethane add 1mL of the GPC standard. Set to volume with dichloromethane, cap, and invert three times. Transfer to properly labeled container.

11.0 Sample Collection, Preservation, Shipment and Storage

- Store sample extracts at 4°C in sealed glass containers. Samples must be completely analyzed within 40 days of extraction. Also, see SOP NE027.

12.0 Quality Control

- The analyst should demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples.

- For the samples sets, the associated QC samples must be processed through the GPC as well.

- The dichloromethane is degassed prior to use by the GPC by purging with nitrogen. Also, avoid extreme ambient temperatures as that may contribute to solvent out-gassing, which can lead to retention time shifts.

- The retention times for bis(2-ethylhexyl) phthalate and perylene must not vary more than $\pm$ 5% between calibrations. If the retention time shift is > 5%, take corrective action.

- The pump flow rate should be periodically checked to ensure the flow rate is correct.

  - Unscrew the solvent line, after the pump but before the GPC, set the pump flow rate to 5mL/min and turn the pump on. Using a 50 or 100mL graduated cylinder collect the solvent in the graduated cylinder and observe the time needed to collect 25mL. If corrections are necessary the pump calibration may be adjusted from the solvent pump menu on the Accuprep™ desktop. If the pump needs significant calibration each week, corrective action must be taken to ensure flow reliability, as it is critical to analyte separation and collection.

- Periodically it's a good idea to run blank solvent through the column with a standard method and to analyze the results to check for contamination of the GPC system.

13.0 Calibration and Standardization

- An injection of 2.5mL of prepared GPC standard must be analyzed minimally once a week to ensure the column is functioning properly (Figure 1). It is also necessary to keep the collection/dump times accurate to ensure proper recovery of target analytes. The UV chromatogram of the injection using the Waters column or the S-X3 column must meet the following criteria:
    - The peaks must be observed and should be symmetrical for all compounds in the calibration solution.
    - Corn oil and phthalate (DEHP) peaks must exhibit >85% resolution.
    - Phthalate (DEHP) and methoxychlor peaks must exhibit >85% resolution.
    - Methoxychlor and perylene peaks must exhibit >85% resolution.
    - Perylene and sulfur peaks must not be saturated and exhibit >90% resolution.
- When calibrating for Organochlorine Pesticides/PCBs, the collection/dump times should be updated in the method to be used to reflect the following:
    - Determine the elution times for the phthalate, methoxychlor, perylene and sulfur.
    - Choose a dump time which removes >85% of the phthalate, but collects >95% of the methoxychlor.
    - Stop collection after the elution of perylene, but before sulfur elutes.
- When calibrating for semi-volatiles, the collection/dump times should be updated in the method to be used to reflect the following:
    - Initiate collection just before the elution of the phthalate, and after the elution of corn oil.
    - Stop collection after the elution of perylene, but before sulfur elutes.

14.0 Procedure

- Prepare the caps to be used with the autosampler vials by inserting the cap liners into the caps, set them aside. (Prepare as many as needed).
- Remove sample extracts from the refrigerator and allow to warm up to room temperature to insure all compounds are dissolved in the solvent.

- Prepare vials for the autosampler. Number them according to their NEA sample ID numbers and put them aside. Prepare the turbo tubes that will collect the samples. Label them according to their NEA ID number also and place a piece of aluminum foil (approx. 3in. X 3in.) over the top of the tubes. This is to avoid contamination in the tubes and also to avoid evaporation of the finished samples; the autosampler needle easily pierces the foil.

- Observe the samples to be analyzed. If there are large amounts of aggregated fats use the centrifuge to try and isolate the solvent. This procedure may not work for all lipids due to the non-polarity of the solvent.

- All samples should be filtered through a syringe filter before being injected onto the GPC system. The Acrodisc filters screw onto the bottom of the Normject disposable syringes. Place the syringe and filter on top of the autosampler vial to be prepared. Remove the plunger; pour the sample into the syringe (8-10 mL of sample while trying to avoid solids and aggregated fats). Reinsert the plunger and depress so the sample is forced through the filter into the vial. Attach the prepared vial cap w/liner to the vials and place numerically in the vial rack. Properly dispose of the syringe and filter. Repeat for all samples and QC in the sample batch.

- Open the AccuPrep™ software and create the sequence for the run. Save the file as the date, followed by a description (i.e., '070106runA.seq' would be the second sequence on 7/01/06).

NOTE: Make sure the solvent reservoirs are full and the purge bottle is empty before beginning an overnight sequence. A standard solvent bottle has a four-liter capacity, at 5 mL/min that will yield approximately 12 samples using the S-X3 pesticides column and 24 samples using the Waters column.

- Begin the sequence.

- Once the sequence has executed, using windows explorer, move the sequence file to 'C:\Program Files\J2 Scientific\AccuPrep305E0\data', and open the AccuChrome™ software.

- Import the data from the AccuPrep305E0\data folder and view the database. Print a sequence report for the run and print a copy of the Calibration Report. Place the reports in the folder and the extraction lab will continue with further clean-up before analysis.

15.0 Calculations

- Calculations for pump speed, UV trace resolution, peak separation, etc. are handled through the AccuPrep™ and AccuChrome™ software.

- For SVOC analysis it should be remembered that only half of the sample extract is processed by the GPC (5 mL of the 10 mL extract is loaded onto the GPC column and concentrated to 1 mL), and thus, a dilution factor of 2 must be used for quantitation of the sample in the determinative method. For PCB/pesticide analylsis (5 mL of the 25 mL extract is loaded onto the GPC column and concentrated back to 5 mL), and thus, no dilution factor is needed in the determinative method.

16.0 Method Performance

- There is no specific method performance associated with this method other than weekly column calibration must be maintained to use this method on sample or QC extracts.

17.0 Pollution Prevention

- See SOP NE168.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

- N/A

19.0 Corrective Action for Out-Of-Control Data

- If peak resolution is unacceptable when inspecting the results of a standard, an effective method of resolving the issue is to back flush the column. Remove the column from the GPC system and install it on a solvent pump with a purge bottle following these steps:
    - Using an approximately 50/50 mixture of dichloromethane and toluene, backward flush the column at 5 mL/min for the remainder of the work day.
    - Slow the pump speed to 1 mL/min overnight.
    - In the morning, ramp the speed back up to 5mL/min for approx 4 hours. Replace the 50/50 mixture with 100% dichloromethane. Flush at 5 mL/min backwards for the remainder of the day.
    - Slow the pump speed to 1 mL/min overnight.
    - In the morning, switch the solvent flow to forwards through the column and run the pump at 5 mL/min for several hours.
- At this point the column should be ready for use. When running toluene, be sure to remove the column from the GPC system. If resolution is still not what it should be, the column may need to be repacked. The Waters high pressure columns are unable to be repacked. The glass pesticides/PCB column should be repacked according manufacturers instructions.

20.0 Contingencies for Handling Out-Of-Control or Unacceptable Data

- N/A

21.0 Waste Management

- See SOP NE054, NE083, NE089.

22.0 References:

1. Wise, R.H.; Bishop, D.F.; Williams, R.T.; Austern, B.M. "Gel Permeation Chromatography in the GC/MS Analysis of Organics in Sludges"; U.S. EPA Municipal Environmental Research Laboratory: Cincinnati, Ohio 45268.

2. Czuczwa, J.; Alford-Stevens, A. "Optimized Gel Permeation Chromatographic Cleanup for Soil, Sediment, Waste and Waste Oil Sample Extracts for GC/MS Determination of Semivolatile Organic Pollutants, JAOAC, submitted April 1989.

3. Marsden, P.J.; Taylor, V.; Kennedy, M.R. "Evaluation of Method 3640 Gel Permeation Cleanup"; Contract No. 68-03-3375, U.S. Environmental Protection Agency, Cincinnati, Ohio, pp. 100, 1987.

4. Test Method, Method 3640; United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory Cincinnati, OH 45268.

23.0 Tables, Diagrams, Flowcharts and Validation Data

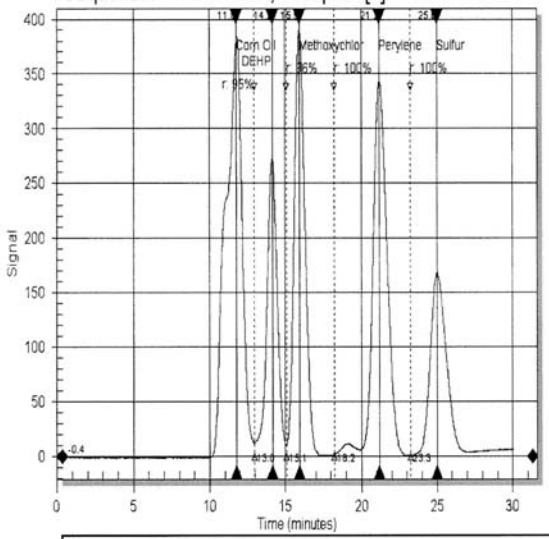
Print Date: 9/28/2005
Print Time: 14:04:31

Column Calibration Report

Sample ID: 062705calA
Sequence: 062705calA
Description: sample
Volume: 2500
Method: svoa012705
Position: 1
Outcome: Sample processed normally

Start Time: 10:14:52, 06/27/05
End Time: 10:49:46, 06/27/05
Column Name: WAT036554
Description: WATERS ENVIROGEL
Serial #: M00275D 02
Pressure Limit: 350

Sequence: 062705calA, Sample: [1] 062705calA



062705calA

Peak	Sample	Retention Time	Pk Ht	Base	Res.	Tol.	P/F
1	Corn Oil	11.8	383	-0.4			
2	DEHP	14.2	272	-0.4	95.5	85.0	P
3	Methoxychlor	15.9	0	-0.4	96.2	85.0	P
4	Perylene	21.2	344	-0.4	99.6	85.0	P
5	Sulfur	25.0	167	-0.4	99.8	90.0	P

Figure 1

SOP NE226_01

REVISION INFORMATION

Version	Revision	Date Effective	Name/Title	SOP Status
Ne226_01	01	01/08/08	Roy Smith Jr., SVOC Manager	Reviewed
			Robert E. Wagner, Laboratory Director	Reviewed
			Christina L. Braidwood, QC Officer	Approved

STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

NE227_02.SOP
REVISION NUMBER: 02

Standard Operating Procedures for Sample Receipt, Sample Storage and Security

JUNE 7, 2007

COPY #__

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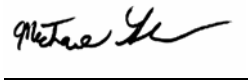
STANDARD OPERATING PROCEDURE

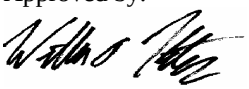
Author: Lenore Nelson
Sample Receiving Dept. Manager

Reviewed by:



Michael Glenn
Lab Supervisor

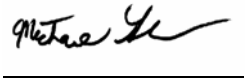
Approved by:



William Kotas
QA/QC Officer

Northeast Analytical, Inc.
Issuing Section: Sample Receiving
SOP Name: NE227_02.DOC
Date: June 7, 2007
Revision: 02

NELAP Annual Review
Date last reviewed: June 7, 2007
Reviewed by: Michael Glenn



Lab Supervisor

1.0 Title

Standard operation procedures for sample receipt, sample storage and sample security.

2.0 Applicable matrix and Matrices

This SOP is applicable to any/all Matrices received at Northeast Analytical.

3.0 Detection Limits

N/A

4.0 Scope

This SOP applies to all samples received for analysis, subcontracting or archival storage.

5.0 Summary

5.1 The temperature of the samples is taken.
5.2 The COC is signed.
5.3 If necessary, sample pH is checked.
5.4 Samples are stored in the appropriate refrigerator/freezer.

6.0 Definitions

N/A

7.0 Interferences

N/A

8.0 Safety

Safety glasses must be worn in the laboratory at all times. During sample handling, disposable gloves should be worn. Caution should be used whenever opening coolers and handling samples.

9.0 Equipment and Supplies

9.1 Cole-Parmer Standard Model #39650-02 Laser Thermometer.
9.2 Traceable Model # 61220-416 Digital Thermometer or equivalent.
9.3 ColorpHast indicator strips
9.4 Disposable transfer pipettes
9.5 Sample Preservation Logbook

10.0 Reagents and Standards

N/A

11.0 Sample Collection, Preservation, Shipment, and Storage

See SOP NE060.doc

12.0 Quality Control

N/A

13.0 Calibration and Standardization

N/A

14.0 Procedure

14.1 Sample Receipt

14.1.1 Recording Sample Temperature

14.1.1.1 Acceptable temperature range for most samples is 1-6(^{o}C). Samples that are hand-delivered to the laboratory immediately after collection may not meet the above criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice. If the sample was recently taken and chilled, but is outside of this range, make note of it on the COC by circling "chilled" and noting the actual temperature in the space provided

14.1.1.2 Check for the presence of a temperature blank with the samples. If one is present, use the digital thermometer. If there is not a blank, use the laser thermometer.

14.1.1.3 To use the digital thermometer, set the temperature blank on the table. Insert the probe into the temperature blank and hold, being careful not to touch the sides of the container. Wait for the digital readout to stop

changing and record the temperature in degrees C on the chain of custody (COC). The temperature blank should be discarded after taking the reading. *Note: The thermometer must have a current calibration check label. The thermometer calibration is verified annually against a* NIST traceable thermometer.

14.1.1.4 To use the laser thermometer, aim approximately six inches away from a sample label. The thermometer should be pointed at sample label to avoid refractive corrections caused by I.R. measurements of clear or reflective surfaces. *Note: The laser thermometer must have a current calibration check label. The thermometer calibration is verified annually against a NIST traceable thermometer. The Laser Thermometer calibration is also checked daily against a refrigerated temperature blank.*

14.1.1.5 If samples shipped to the laboratory are received outside temperature requirements notify client and ascertain whether analysis of samples should be performed or whether re-sampling and is required. If available, consult the appropriate project specific quality assurance sampling plan to determine corrective action to be taken. Temperature measurements are recorded on the sample delivery group Chain of Custody and are transmitted to the client with the final data reports.

14.1.2 Samples Delivered by Client

14.1.2.1 Inspect all samples for leakage or breakage.

14.1.2.2 Water samples for the volatiles analysis should be inverted to check for head-space exceeding a pea sized bubble (approximately 1cm diameter). Any observable headspace should be noted.

14.1.2.3 Record the temperature of the samples on the COC. (See 14.1.1 above)

14.1.2.4 Immediately inform the client of any problems with the samples.

14.1.2.5 Verify the COC against the samples to make sure everything is correct. If the client does not have a COC filled out, they need to do so before departing the laboratory.

14.1.2.6 Any corrections that need to be made should be initialed by the client when they make the changes.

14.1.2.7 After the client and the NEA personnel receiving the samples have signed the COC, and copy should be made and given to the client to keep for their records.

CHAIN OF CUSTODY RECORD PAGE __ 1 OF 1 _

NORTHEAST ANALYTICAL, INC.
2190 Technology Drive, Schenectady, NY 12308
Telephone (518) 346-4592 Fax (518) 381-6055
www.nealab.com information @nealab.com

LRF # __ <06040090>__

DISPOSAL REQUIREMENTS: (To be filled in by Client)
- ○ RETURN TO CLIENT
- ● DISPOSAL BY NORTHEAST ANALYTICAL
- ○ ARCHIVAL BY NORTHEAST ANALYTICAL

Additional charges incurred for disposal (if hazardous) or archival. Call for details.

CLIENT (REPORTS TO BE SENT TO): NORTHEAST ANALYTICAL	PROJECT#/PROJECT NAME: MDL STUDY
PROJECT MANAGER: BOB STOLL	LOCATION (CITY/STATE) ADDRESS: N/A
SAMPLED BY: (Please Print) N/A	REQUIRED TURN AROUND TIME: 1 MONTH
SAMPLING FIRM:	NAME OF COURIER (IF USED):
ELECTRONIC RESULTS	
FAXED RESULTS	FAX #:

ENTER ANALYSIS AND METHOD NUMBER REQUESTED

PRESERVATIVE CODE:
BOTTLE TYPE:
BOTTLE SIZE:

PRESERVATIVE KEY: 0 - ICE; 1 - HCL; 2 - HNO3; 3 - H2SO4; 4 - NaOH; 5 - Zn. Acetate; 6 - MeOH; 7 - NaHSO4; 8 - Other (Na2SO3)

SAMPLE ID	DATE	TIME	MATRIX	GRAB/ COMP	LAB SAMPLE ID (NEA USE ONLY)	NUMBER OF CONTAINERS	AROCLOR 1248	REMARKS:
TO-10A LL 1248 #1	4/21/06		AIR/LL		AJ04300		x	
TO-10A LL 1248 #2	4/21/06		AIR/LL		AJ04301		x	
TO-10A LL 1248 #3	4/21/06		AIR/LL		AJ04302		x	
TO-10A LL 1248 #4	4/21/06		AIR/LL		AJ04303		x	
TO-10A LL 1248 #5	4/21/06		AIR/LL		AJ04304		x	
TO-10A LL 1248 #6	4/21/06		AIR/LL		AJ04305		x	
TO-10A LL 1248 #7	4/21/06		AIR/LL		AJ04306		x	
TO-10A LL 1248 #8	4/21/06		AIR/LL		AJ04307		x	
								Note:Final Effluent PCB sample is on hold pending results of Final Effluent Composite sample

AMBIENT OR CHILLED: TEMP: COC TAPE: Y N PROPERLY PRESERVED: Y N OTHER NOTES:
RECEIVED BROKEN OR LEAKING: Y N COC DISCREPANCIES: Y N RECVD W/I HOLDING TIMES: Y N

RELINQUISHED BY	RECEIVED BY	RELINQUISHED BY	RECEIVED BY	RELINQUISHED BY	RECEIVED BY
SIGNATURE	SIGNATURE	SIGNATURE	SIGNATURE	SIGNATURE	SIGNATURE
PRINTED NAME	PRINTED NAME	PRINTED NAME	PRINTED NAME	PRINTED NAME	PRINTED NAME
COMPANY	COMPANY	COMPANY	COMPANY	COMPANY	COMPANY
DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME

S:\LOGIN\MDL\COCS

14.1.3 Samples Received by Delivery Service

14.1.3.3 Upon delivery, check the package for any apparent damage caused during shipping.

14.1.3.4 Open package and remove contents. If there is a suspected leak open the package in a fume hood.

14.1.3.5 Record the temperature of the samples. (See 14.1.1 above)

14.1.3.6 After all the samples are unloaded onto the table, verify against the COC.

14.1.3.7 The COC should be signed by NEA personnel with the date and time that samples were dropped off by the delivery service.

14.1.3.8 If any of the samples are broken or leaking, if the temperature is too high, or if there are any discrepancies with the COC the client needs to be notified immediately.

14.1.3.9 The details of the phone call or e-mail correspondence must be documented on a phone log or e-mail printout and put in the LIMS notes. All correspondence documentation is maintained with COC

records.

14.1.4 Low Level PCB Congener/PCB 8082 Samples

14.1.4.1 The designated low-level samples must be checked in by a technician from the water lab to avoid PCB sample cross-contamination.

14.1.4.1 The technician brings their own supplies including a transport cart, acetone, gloves, and paper towels.

14.1.4.2 Coolers are opened with a paper towel saturated with acetone being careful not to touch the cooler.

14.1.4.3 The technician removes the samples from the cooler and places them on their cart.

14.1.4.4 Record the temperature of the samples on the COC. (See 14.1.1 above)

14.1.4.5 After all samples have been removed from coolers, technician then reviews and verifies the sample IDs on the COC against the sample labels.

14.1.4.6 The Water Lab Technician then removes the samples from the sample receiving area and places them in storage. Samples received for any analysis besides low-level PCB Congener/PCB 8082 will then be the responsibility of the Sample Custodian.

14.1.5 Checking the pH

14.1.5.1 A pH measurement of aqueous samples requiring preservation must be performed in a hood upon sample receipt for non-volatile samples. *Note: the pH of aqueous samples received for volatiles analysis is recorded by the volatiles section analyst.*

14.1.5.2 Using a disposable pipette, a small aliquot of sample should be pulled from the sample.

14.1.5.3 The pipette should then be emptied over a pH strip, which should be over a disposable cup.

14.1.5.4 The disposable cup should be emptied into the appropriate waste stream and the cup disposed of.

14.1.5.5 Make sure to preserve the sample if it is not at the correct pH, using the proper preservative.

14.1.5.6 The results should be recorded in the preservation logbook located in sample receiving. (see attachment II for preservation logbook)Be sure to note if preservative needed to be added.

14.1.6 Sample Acceptance Policy

Northeast analytical maintains a written sample acceptance policy

available to NEA clients. In general, samples will not be accepted if they present hazards that cannot be adequately controlled by the laboratory for example biohazards and known hazardous radioactive samples. In addition, appropriate sample containers should be used for each analysis and specified sample hold times should be met. Please see Attachment III for sample acceptance policy. If a client requests samples be analyzed that do not meet hold time or container requirements the associated sample analysis reports are flagged to denote the non-compliance and a sample delivery group case narrative may be issued as necessary.

14.2 Sample Storage

14.2.1 Volatiles Samples

14.2.1.1 Volatiles soil and water samples are stored separately from other samples to decrease the possibility of cross-contamination. All of volatiles refrigerators are in VOA storage, room 120.

14.2.1.1 Soil and solid samples are stored in R-9.

14.2.1.2 Water and liquid samples are stored in R-7.

14.2.1.3 Trip blanks associated with soil samples should be stored in R-9 with the soils.

14.2.2 Soil Samples

14.2.2.1 All other soil and solid samples are stored in R-5 on the appropriate shelf.

14.2.2.2 Samples to be extracted by the main solid extractions lab will be placed on the shelves in the middle of the refrigerator.

14.2.2.3 Samples to be extracted only by the metals lab will be placed in the far left corner.

14.2.3 Water Samples

14.2.3.1 All waters except for volatiles and low level PCB Congener/8082 are stored in R-5 on the appropriate shelf.

14.2.3.2 All waters to be extracted by the water lab go on the shelves directly to your right as you enter R-5.

14.2.3.3 Waters to be prepared by Metals Department are placed on the shelves against the left wall of R-5.

14.2.4 Low Level PCB Congener/8082 Samples

14.2.4.1 All low level PCB congener/8082 samples should be stored in R-20C.

14.2.4.2 These samples should only be handled by a technician from the water lab.

14.2.5 High level PCB DNAPL samples

14.2.5.1 Samples of PCB DNAPL (neat PCB and high percentage PCB liquids) are stored in the waste room high level PCB refrigerator R15. (not temperature monitored).

14.2.6 Biota tissue samples and archived frozen samples

14.2.6.1 Samples of biota tissue and frozen archived samples are stored in the walk-in deep freezer R4.

14.3 Refrigerator/Freezer Monitoring

14.3.1 Storage Area Temperature monitoring

14.3.1.1 All refrigerators and freezers used for sample storage are monitored and recorded twice per day by sample login department personnel during lab business days. (please see Attachment I -Temperature Logbook) If temperatures are outside stated limits on logbook the unit should be checked for possible thermostat adjustment, door ajar, frequent usage by lab personnel or need for defrosting. The temperature should be checked again within 2-3 hours when excursion occurs. If temperatures are still out of limits consult QA department or management for refrigerator servicing. . *Note: The thermometer must have a current calibration check label. The thermometer calibration is verified annually against a NIST traceable thermometer.*

14.3.2 General Monitoring

14.3.2.1 All storage areas should be periodically inspected for sample leakage, unusual odors and general neatness. Consult QA department, Sample Login Manager or Lab Management if sample spills occur.

14.4 Sample Security

14.4.1 General Sample Security

14.4.1.1 Sample storage areas are restricted to NEA personnel. The main sample storage area R5 and R12 is locked after business hours. Access to the laboratory building is restricted by electronic key card after business hours Monday -Friday 8:00 AM-5:00 PM.

14.4.2 Litigation Sample Security

14.4.2.1 If samples are received from projects that require formal chain of custody and high level security for legal purposes or other purposes, the samples are stored in padlocked storage lockers inside the cooler or in a padlocked refrigerator. When samples are removed from storage and returned to storage for processing an Internal Chain of Custody log is completed. All transfers of custody between laboratory personnel are recorded on the Internal Chain of Custody. Please see Attachment 23.3 for example Internal Chain of Custody Form.

15.0 Calculations

N/A

16.0 Method Performance

N/A

17.0 Pollution Prevention

N/A

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

N/A

19.0 Corrective Action for Out-of-Control Data

N/A

20.0 Contingencies for Handling Out-of-Control of Unacceptable Data

N/A

21.0 Waste Management

Please see NE054.SOP, NE083.SOP, and NE089.SOP

22.0 References

22.1 NYS-DOH ELAP Manual

22.2 NELAC Quality Systems Document Rev. 2003

23.0 Tables, Diagrams, Flowcharts and Validation Data

23.1 Refrigerator Temperature Logbook

23.2 Sample Preservation Logbook

23.3 Internal Chain of Custody

23.4 Sample Acceptance Policy

23.1 Refrigerator Temperature Logbook

Northeast Analytical Inc.

Refrigerator / Freezer Temperature Logbook
UNIT # 5

File: S:\FORMS\LOG\TEMP\TEMP_LOG.xls

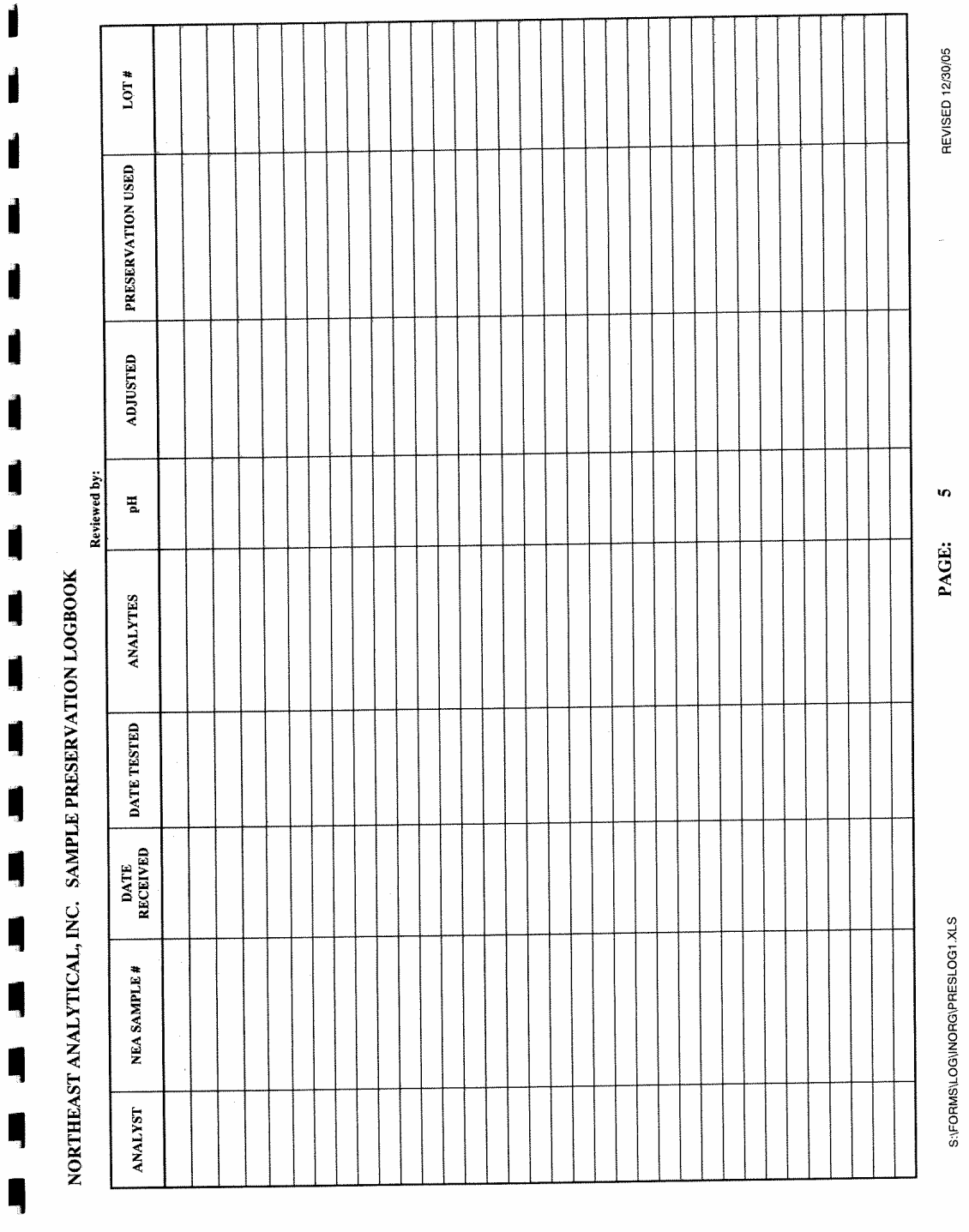
Date	AM Temp	AM Time	AM Initials	PM Temp	PM Time	PM Initals	Comments
13-Jul-06	3.0	800	MC	3.0	415	LN	
14-Jul-06	2.5	800	MC	3.0	415	MC	
15-Jul-06							
16-Jul-06							
17-Jul-06	2.5	800	MC	3.0	415	MC	
18-Jul-06	2.5	800	MC	2.5	415	MC	
19-Jul-06	3.0	800	LN	3.0	430	LN	
20-Jul-06	3.0	800	MC	2.5	445	MC	
21-Jul-06	2.5	800	MC	2.5	430	MC	
22-Jul-06							
23-Jul-06							
24-Jul-06	2.5	800	MC	2.5	445	MC	
25-Jul-06	2.5	800	MC	2.5	415	MC	
26-Jul-06	3.0	800	LN	3.0	445	LN	
27-Jul-06	2.5	800	MC	2.5	430	MC	
28-Jul-06	2.5	800	MC	2.5	430	MC	
29-Jul-06							
30-Jul-06							
31-Jul-06	2.5	800	MC	3.0	415	MC	
01-Aug-06	2.5	800	MC	2.5	445	MC	
02-Aug-06	3.0	800	LN	3.0	500	LN	
03-Aug-06	2.5	800	MC	2.5	430	MC	
04-Aug-06	3.0	800	MC	3.0	430	MC	
05-Aug-06							
06-Aug-06							
07-Aug-06	2.5	800	MC	2.5	400	MC	
08-Aug-06	2.5	800	MC	3.0	430	MC	
09-Aug-06							

Comments: _____

Refrigerator Temperature Limits: >0°C to 6°C
Freezer Temperature Limits: (SEE LABEL ON UNIT)

PAGE: 8

23.2 Sample Preservation Logbook



NORTHEAST ANALYTICAL, INC. SAMPLE PRESERVATION LOGBOOK

Reviewed by:

ANALYST	NEA SAMPLE #	DATE RECEIVED	DATE TESTED	ANALYTES	pH	ADJUSTED	PRESERVATION USED	LOT #

S:\FORMS\LOG\NORG\PRESLOG1.XLS

PAGE: 5

REVISED 12/30/05

23.3 Internal Chain of Custody Example

Northeast Analytical Inc.
2190 Technology Drive
Schenectady, NY 12308

Internal Chain of Custody Transfer

Sample Delivery Group: _____ **(FOR SAMPLES ONLY)** Department _____
NEA ID #: _____
Received from client by: _____ Date: _____ Time: _____
Storage Location : _____

Received from:	Date:	Time:	Relinquished to:	Date:	Time:	Comments:/Sample Storage Location:

Form: S:\FORMS\CATBIN_COC_S.WK4 (rev1, 8/26/99)

Page___ Of ___

23.4 Sample Acceptance Policy Statement

NORTHEAST ANALYTICAL, INC

Sample Acceptance Policy

Northeast Analytical is a New York State Department of Health Certified Lab (NYS-DOH -ELAP) and is also certified by the National Environmental Lab Accreditation Council (NELAC). ELAP/NELAC requires that laboratories maintain a written policy for the acceptance of samples. Samples submitted to Northeast Analytical must meet the following criteria in order to be processed and analyzed in an accurate and timely manner.

A. **Chain of Custody (COC)**

All samples arriving at the lab should be accompanied by a COC. Information on the COC should be printed legibly and must be filled out completely.

This includes:

1. The name of the company and project manager who will be receiving results.
2. Contact information including phone number and address.
3. A specific project name if one is to be designated.
4. The location where the samples were taken.
5. Sampler information.
6. Required turn around time. (Standard is 10 business days.)
7. A unique sample ID, (one per line) for each sample taken.
8. Sampling information: date, time, matrix, grab vs. composite, and number of containers.
9. The requested analysis, (one per line) with the appropriate boxes marked to match up sample and the analyses it should be tested for.
10. Disposal requirements. Unless otherwise specified all samples will be disposed after 45 days after receipt at the lab.

The COC should also be signed at the bottom to signify the samples were relinquished to the lab. If the samples are hand delivered, the courier/sampler should sign the COC upon arrival at the lab to relinquish sample. If the samples are shipped, they should be relinquished by the client before pick up by UPS/Fed Ex/DHL or other shipper.

B. Condition of the Samples

All samples should be chilled during transportation to the lab. Samples will be considered acceptable if they fall in a range of 0-6 degrees C. Samples that are hand delivered immediately after collection will most likely not meet that criterion. In this case, the samples will be acceptable if there is evidence that the chilling process has begun. (for example: ice is present in cooler)

The samples should be in the appropriate sample container with enough material for the analysis. For assistance in selecting containers and required sample amounts please feel free to contact the lab.

If placing a bottle order, a minimum of 24 hours notice should be given if possible.

Each sample container should have a dry and legible label attached. The information on the label should match the COC and include the company name, preservatives used in the container, and the information listed in #s 4 and 7-9 above.

Please Note: Any discrepancies between the label and the COC may slow down the processing of the sample.

EPA has specified holding times for samples based on the analysis requested. Please be aware of these when sampling so that the lab will receive the samples with enough time to perform all the required analyses. If samples are received past holding times and analysis is still requested the final data reports will be flagged to denote the excursion.

Common Sample Hold Times in Days from Date of Collection.								
Test	EPA 8082/608	PCB	EPA 8081/608	EPA 8270	pH	EPA 8260/624/602	EPA 8151	Total Residue
Matrix	PCBs	PCB Congeners	Pesticide	SVOC		Volatiles	Herbicides	Solids
Water	7	5	7	7	ASAP	14*	7	7
Solid	14	14	14	14	14	14	14	X

* Preserved samples. Unpreserved have a 7 day hold time.

If a rush turn around time is requested the lab must be notified ahead of time to guarantee that the rush turn can be met. If the samples arrive at the lab with no prior notice the analysis may not be completed on time.

Thank you for choosing Northeast Analytical Inc. We appreciate your cooperation in following these guidelines. If you have any questions of comments please feel free to contact the lab at:

Northeast Analytical Inc.
2190 Technology Drive
Schenectady, NY 12309
Ph (518) 346-4592
Fax (518) 381-6055
www.nealab.com

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

LABORATORY SERVICES SECTION

QUALITY ASSURANCE PROGRAM PLAN

May 5, 2003
Version 2.0, rev 0

Prepared By:
Bob Avery, Laboratory Director
Laboratory Services Section
Environmental Sciences and Services Division
Michigan Department of Environmental Quality

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http://www.deq.state.mi.us/dwr/Lab/Lab.htm

Quality Manual Signature Page
MDEQ Laboratory Services Section
Version 2.0 Revision: 0

LABORATORY SERVICES SECTION QUALITY ASSURANCE PLAN FOR MICHIGAN DEPRATMENT OF ENVIRONMENTAL QUALITY Version 2.0 REVISION 0

EFFECTIVE DATE
May 5, 2003

Prepared by: Bob Avery
3350 N. ML King Jr. Blvd.
Lansing, MI 48906
(517) 335-9800

Bob H. Avery	**Bob Avery**	**Laboratory Director**	May 12, 2003 Date
Sandra K Gregg	**Sandra Gregg**	**Inorganic Unit Technical Manager**	5-12-2003 Date
Carol Smith	**Carol Smith**	**Organic Unit Technical Manager**	5-12-03 Date
Sandra Kerns	**Sandra Kerns**	**Drinking Water Unit Technical Manager**	5/12/2003 Date
George K Krisztian	**George Krisztian**	**Laboratory Compliance Officer**	5/12/2003 Date
Albert Lee	**Albert Lee**	**Quality Assurance Officer**	5/12/2003 Date

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Table 1 MDEQ Environmental Laboratory Capabilities and Reporting Limits

Figure 2-1 Organization chart

1.0 POLICY STATEMENT

1.1 This Quality Assurance Manual summarizes the policies and operational procedures associated with the MDEQ Laboratory Services Section at 3350 N. ML King Blvd., Lansing Michigan. Specific protocols for sample handling and storage, chain-of-custody, and laboratory analyses, data reduction, corrective action, and reporting are described. Further details on these policies and procedures are contained in SOPs and related documents. This Quality Manual, Laboratory SOPs, and related documentation describe the quality system for the MDEQ Laboratory.

The laboratory performs bacteriological analyses in drinking water, surface water and wastewater, as well as chemical analyses for inorganic and organic constituents in water, air, oil, and solids for various state and federal programs. The Laboratory's goal is to produce the highest quality data that is scientifically valid, defensible, and of known and documented quality in accordance with standards developed by the state and EPA regulations or requirements. The Drinking Water Laboratory Certification Program is also administered under the Laboratory Services Section, independent of the analytical laboratory services provided. This program certifies laboratories for chemical and microbiological analysis of drinking water from the state of Michigan. See Appendix E for program manual.

The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. This includes a review of facilities and instrumentation, staffing, and any special QC or reporting requirements to ensure that analyses can be performed within the expected schedule. All measurements are made using published reference methods or methods developed by the MDEQ Laboratory Section.

1.2 Granted Authority

DRINKING WATER LABORATORY

AUTHORITY: The laboratory was created by 1907 P. A. 109, and is in operation based on 1978 P.A. 368, Section 9601, as amended, to maintain and operate laboratories for the protection of the public health. Michigan is required to have a principal state Drinking Water Laboratory certified by USEPA to maintain primacy for the Public Water Supply Supervision program under the Safe Drinking Water Act.

ENVIRONMENTAL LABORATORY

AUTHORITY: The Environmental Laboratory was first started under the direction of the Department of Public Health in 1929 under the Water Resources Commission Act (1929 P.A. 245). The two environmental lab units support environmental enforcement, regulatory, and monitoring programs in MDEQ, to include RCRA, SARA, UST, Clean Air Act, Clean Water Act, and Part 201 of the Natural Resources and Environmental Protection Act, Act 451 of 1994, as amended.

DRINKING WATER LABORATORY CERTIFICATION PROGRAM

AUTHORITY: USEPA granted primacy to the State of Michigan in 1976 for the certification of Drinking Water Laboratories as specified by 40 CFR 142.10b(3) and Safe Drinking Water Act, 1976 P.A. 399, as amended. The state must retain oversight, issue the certificate, and maintain a state laboratory certified by USEPA capable of testing all contaminants listed in the drinking water standards.

1.2 PROGRAM DESCRIPTION

The ESSD Laboratory Services Section is the scientific testing arm of the Department of Environmental Quality. The analytical results produced by the laboratory drive science-based departmental decisions related to health investigations, environmental site cleanup and remediation. It is a not-for-profit, objective, scientific testing laboratory within the department, analyzing samples for the public, local, county, state and federal agencies. The test results from this laboratory are also used for the prevention/detection of public health outbreaks in drinking water, identifying chemical constituents of environmental pollution, and identifying sources of contamination. The laboratory is a key component in the testing of environmental samples from suspected Bioterrorism events.

The Laboratory Services Section has three areas of expertise and focus: the Environmental Laboratory; the Drinking Water Laboratory; and the Laboratory Certification Program. Each area provides a stable workforce of Laboratory Scientists whose knowledge and expertise insure sound scientific testing, court qualified data and expert witness testimony in department initiated criminal and civil litigation. It provides a wide range of scientific testing and review capabilities to the public, local, county, state, and federal agencies to detect/monitor public health outbreaks and environmental contamination. All three areas perform audits of private and public sector laboratories to insure data quality and compliance with state and federal rules. The section serves as a reference laboratory to resolve disputes on sample data submitted to DEQ for enforcement, compliance and monitoring purposes.

- Environmental Laboratory:
    - Testing for many compounds, including Mercury, DDT, PCB's, Ozone testing, particulates
    - Incinerator testing
    - Air, surface water, soil sample testing
    - Brownfield testing for redevelopment
    - Landfill testing for containments & monitor for leakage
    - Identify contaminants in watersheds
    - Provide scientific identification and quantification of contaminated sites
    - Monitor clean-up activities to determine efficacy of remediation
    - Mobile Laboratory – on-site testing

- Drinking Water Laboratory:

    - Provide scientific testing to drinking water supplies to prevent public health outbreaks.
    - Provide available scientific testing to private and public water supplies for EPA/State/County required chemical and biological monitoring.
    - Provide testing for beach monitoring programs that determine wheter to issue swimming advisories.
    - Provide testing for the regulated community and public to monitor swimming pools and spas to insure water quality.
    - Protection of drinking water sources from biological/chemical terrorism

- Laboratory Certification:

    - Insures laboratory integrity for quality testing
    - Identify certified laboratories for public use
    - Investigate complaints about certified laboratories
    - Monitor certified laboratories to insure compliance to regulations through review of sample proficiency testing program evaluations.
    -

NELAC: The Laboratory Director and Laboratory Certification Officer actively participate on the National Environmental Laboratory Accreditation Conference (NELAC). Both staff members work with other state and federal representatives to draft and vote on national standards for the certification of laboratories.

BIOLOGICAL/CHEMICAL TERRORISM: The section participates with the State Emergency Management System bioterrorism execises to prepare for biological/chemical attack response. The lab coordinates analytical testing with the other state laboratories, including DCH, MSP, MDA, and CIS. The laboratory has capability to test air, drinking water, surface water, and soil samples.

AUDITS: The laboratory staff perform comprehensive on-site audits for the department to insure data integrity and contract specifications. DEQ has several Overflow Contract Laboratories for testing of samples in the event the DEQ Laboratory does not have capacity during the heavy sampling season or for specialized testing that cannot be done at the state laboratory. This in-depth audit reviews test sample data, test methods, data documentation, data storage, chain-of-custody, and includes discussions with the analytical staff for method compliance.

ENFORCEMENT: The laboratory serves the Office of Criminal Investigation for testing samples under criminal or civil investigation. The staff also serve as a stable source of "expert witnesses" for the prosecutors and the Office of the Attorney General for prosecution and/or cost recovery.

DATA REVIEWS: In addition to sample testing, the laboratory also reviews private and public sector testing data. In many instances, test data from other laboratories may be subject to question due to split sample comparisons or inaccuracies of the final report. The state laboratory will review the test methods and raw data for any problems such as changes to the test method, time shifting, sink testing, poor quality control, or peak shaving. A report will be issued to accept the data, investigate further for criminal action, or reject the data.

ALTERNATE TEST PROCEDURE (ATP) REVIEWS: Many laboratories request the use of alternate test methods that are not promulgated/listed by EPA or the state. These methods, technology, instruments, and quality control must be reviewed to insure data quality. The laboratory will issue a report either recommending that EPA accept the ATP request or reject the request.

STATE AND FEDERAL ASSISTANCE: The laboratory staff provides assistance to the other state and federal agencies. Examples include auditing the MN state Drinking Water Laboratories for EPA when they did not have a certified Microbiology Auditor available; auditing the Mt. Pleasant Tribe Drinking Water Laboratory for EPA; assisting DOT and MSP on a Joint Evaluation Committee to select a Laboratory Information Management System (LIMS), EPA criminal investigations, drinking water testing for MDA, and testing of environmental samples from suspected methamphetamine laboratories.

TRAINING: The laboratory staff provide scientific training to the department staff. Topics include Laboratory Analytical Testing, Detecting Problems with Analytical Data, Laboratory Enforcement Training, Limits of Detection, and Proper Sample Collection for Analytical Testing.

FACILITIES: Three facilities are staffed by this section: the State Laboratory Building at 3350 N. ML King, the Program Support Facility at 815 Filly Street, and the State Laboratory Annex at 815 Terminal Street.

- The **State Laboratory Building** houses the majority of the testing equipment and staffing. The building is not a public facility and provides 24/7 security. The Department of Community Health Laboratory and the DEQ Laboratory share this building to provide clinical and environmental testing capability to the state.

- The **Program Support Facility** provides specialized air quality testing (ozone, paint, coal/oil fuels), equipment configuration and monitoring. The laboratory staff assigned to this facility provide equipment/instrument repair, custom fabrication of monitoring equipment/trailers, and equipment maintenance for the Air Quality Division programs. The Mobile Laboratory is stationed here between on-site scheduling. The building also houses the SWQD Toxicity Testing Laboratory and the Waste and Hazardous Materials Division Soil Testing Laboratory. Laboratory chemical solvents are stored in a specially built solvent storage shed attached to the building. Boats and other surface water collection equipment from SWQD are stored in the garage areas. This facility serves as the main storage facility for DEQ for items that cannot be stored at Constitution Hall.

- The **State Laboratory Annex** provides specialized testing and also serves as the storage area for the laboratory's chemicals and supplies as well as the staging area for sample kit preparation for sample collection.

2.0 ORGANIZATION AND RESPONSIBILITIES

2.1 Organization Chart

An organization chart for the MDEQ – ESSD Laboratory is shown in Figure 2-1.

The Section Chief is the Laboratory Director for the Laboratory Services Section (LSS), and manages the Environmental Laboratory, the Drinking Water Laboratory, and the Laboratory Certification Program. There are four units in the LSS, each managed by a Unit Chief (also known as a Technical Manager). Each Unit may have Senior Level Lead Worker(s) to coordinate the activities of the analysts and technicians. Designated Lead Workers may serve as temporary Acting Unit Chief (Technical Director) in the event a Unit Chief is not available.

2.2 Laboratory Director (Laboratory Services Section Chief)

The Laboratory Director is responsible for:

- Overall management of the laboratory operations including data quality, providing appropriate resources and facilities to perform requested work, and supervision of office and analytical staff;
- Defining the minimal level of experience and skills necessary for all positions in the laboratory and identifying the proper Civil Service classification position and level;
- Providing all appropriate safety items and analytical tools necessary for the proper completion of tests;
- Ensuring that all department, division, and laboratory polices and procedures are properly followed;
- Achieving the federal, state, department, division and laboratory goals and objectives;
- Ensuring timely compliance with audits and corrective actions;
- Maintaining a positive working environment that encourages open, constructive problem solving, continuous improvement and customer service;
- Developing a proactive program for prevention and detection of improper, unethical or illegal actions

2.3 Quality Assurance (QA) Officer

As shown in Figure 2-1, the QA Officer is independent of analytical duties, and has direct access to the Compliance Officer and Laboratory Director, to resolve any dispute involving data quality. The QA Officer serves as the focal point for QA/QC and is responsible in the review of quality control data with the Unit Managers. The officer is responsible for auditing the implementation of the Quality System. The QA Officer has sufficient authority to stop work as deemed necessary in the event of serious QA/QC issues. Specific functions and duties include:

- Conducting internal audits on the entire technical operation annually; and
- Notifying laboratory management of deficiencies in the quality system and monitoring corrective action.

- Review of the MDLs, SO's & IDOCs.
- Monitor the Federal Register to keep current with newly promulgated regulations.
- Reviewing PT data to ensure the laboratory is meeting prescribed objectives and to determine systematic trends.
- Develop and maintain safety protocols in the laboratory to insure compliance with applicable regulations.
- Review QC data to determine if methods are in control or if trends are developing.

2.4 Unit Technical Directors (Unit Managers)

The **Unit** Technical Directors (Unit Managers) for each Unit reports to the Laboratory Director and is responsible for:

- Certifying personnel through an Initial Demonstration of Capability (IDOC) and overseeing all tests performed in the unit;
- Monitoring daily standards of performance in quality control and quality assurance;
- Monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data;
- Ensuring that sufficient numbers of qualified personnel are employed to perform and review the work of the unit; and
- Providing educational direction to laboratory staff;
- Ensuring the data quality and integrity of the analytical testing;
- Providing for the health and safety of staff while working in the facility by developing and maintaining safety protocols to insure compliance with applicable regulations.
- Provide training and serve as a resource for agency and division clients.
- May be designated and serve as temporary Acting Section Chief (Laboratory Director) in the event the Laboratory Director is not available.

2.5 Technical Staff (Laboratory Scientists and Laboratory Technicians)

Technical staff are responsible for preparation and sample analysis, data review, general equipment maintenance, LIMS entry, and identification of corrective actions. The staff report directly to the Unit Technical Directors. All personnel are responsible for complying with all quality assurance/quality control (QA/QC) requirements that pertain to their organizational/technical function. As documented in the employee records, each technical staff member has the experience and education to adequately demonstrate knowledge of their particular function and a general knowledge of laboratory operations, analytical test methods, quality assurance/quality control procedures and records management. The technical staff have the authority to halt analysis at any time the testing appears out of control. Communication with the Unit Manager is a daily requirement.

2.6. Training

Each employee has read, understood, and is using the latest version of the laboratory's SOPs, which relates to his/her job responsibilities. Each employee demonstrates continued proficiency by acceptable performance on Laboratory Control Samples (LCS) or Laboratory Fortified Blanks

(LFB). Training records (e.g., continuing education, participation in technical conferences, internal training activities) are kept with personnel files. Training is an integral part of the Performance Appraisal System and is documented with each employee's annual performance factors and evaluations.

2.7 Laboratory Capabilities

The Laboratory analyzes ground water, surface water, source water, wastewater, air, oil, and solid samples. Table 1 and APPENDIX E list the parameters/analytes measured, test references, and Reporting Limits (RL).

3.0 QUALITY ASSURANCE OBJECTIVES

The overall QA objectives for the MDEQ Laboratory are to develop and implement procedures for laboratory analysis, chain-of-custody, and reporting that will provide results which are of known and documented quality. Data Quality Indicators (DQIs) are used as qualitative and quantitative descriptors in interpreting the degree of acceptability or utility of data. The principal DQIs are precision, bias (accuracy), and MDL's/RL's. DQIs are used as quantitative goals for the quality of data generated in the analytical measurement process. This section summarizes how specific QA objectives are achieved. The specific application of these various activities are contained in the method SOPs.

3.1 Precision

Precision is a measure of the degree to which two or more measurements are in agreement.

Precision is assessed through the calculation of relative percent difference (RPD) and relative standard deviations (RSD) for replicate samples. For inorganic analyses, laboratory precision is usually assessed through the analysis of a sample/sample duplicate pair and field duplicate pairs. For organic analyses, precision is usually assessed through the analysis of matrix spike/matrix spike duplicate (MS/MSD), or laboratory control sample/laboratory control sample duplicate (LCS/LCSD).

3.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference or true value.

Accuracy is assessed by the analysis of QC samples and through the adherence to all sample handling, preservation and holding times. Laboratory accuracy is further assessed through the analysis of MS/MSD, quality control samples, and surrogate compound spikes, as specified in the SOPs.

3.3 Method Detection Limits (MDL) and Reporting Limits (RL)

Method Detection Limits (MDLs) are determined for all target analytes as specified in 40 CFR Part 136, Appendix B. The MDL Study is completed annually or at the time a major change has occurred that would impact the analytical sensitivity. Laboratory grade water or sand will be used for the MDL determination. A specific matrix may be used if: 1) it is uniform in consistency; 2) routinely available to all laboratories in the state of Michigan; and 3) is representative of a matrix routinely submitted to the laboratory.

The MDEQ Laboratory recognizes that it is impractical to routinely report to the MDL for actual samples. Based on the calculated MDL, a Reporting Limit (RL) is determined, using the specific reference method, the MDL spiking concentration, or a concentration based on these factors and

Best Professional Judgement. For this reason, the RL is used as the routine quantitation limit. Typically, it may be at or near the spike concentration used to determine the MDL. In addition, for ease and convenience of reporting, the RL may be: (1) rounded up to the nearest one, two, five, or ten; or (2) rounded to a uniform number for a group of compounds in a gas chromatograph scan. In any case, the RL must be at or above the MDL and have a calibration standard at or below the RL. The reference SOP may take precedence in the selection of the low standard. See Admin SOP 103, "Method Detection Limit (MDL) and Reporting Limit (RL)". Sample results between the RL and the MDL may be reported to the client with a qualification flag indicating a lower confidence in the quantification process.

4.0 SAMPLING PROCEDURES

4.1. Environmental Laboratory sampling is conducted by MDEQ Field staff or staff under contract by state departments. The laboratory will advise sampling staff of the proper collection bottles, preservatives and storage and sample submission as needed. See Appendix C, Sample Bottle and Preservation Form, for details. Refer also to SOP 121, Sampling Techniques and Sample Submittal, and SOP 124, Sample Receiving/Environmental Laboratory.

4.2 Drinking Water Laboratory sampling is conducted by MDEQ Field staff, staff under contract by state departments, municipal water supplies and the general public. See Appendix F, Drinking Water Sampling Procedures Manual.

5.0 SAMPLE HANDLING

This section summarizes policies and practices for sample handling. Further details are contained in SOPs 109, 117, 119, 121, 122, & 124.

5.1 Sample Tracking

The MDEQ Laboratory assigns a unique Laboratory Information Management System (LIMS) generated Work Order Number and Bottle Number to each sample received to ensure that there can be no confusion regarding identity. This unique identification (ID) number is placed on each sample container, subsample and subsequent extracts and/or digestates.

5.2 Sample Acceptance Policy

The Laboratory has written sample acceptance SOPs that outline the circumstances under which samples will be accepted. Data from any samples that do not meet the policy are noted in the laboratory report defining the nature and substance of the variation. The SOP's require or establish:

- All samples must be from the department, other state agencies or government agencies or staff under the direct supervision and contract of government agencies (no environmental samples will be accepted or tested from the general public);
- The Drinking Water Laboratory accepts samples from the general public for fee testing.
- Proper, full, and complete documentation, including the sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;
- Unique identification of samples using durable labels completed in indelible ink;
- Use of appropriate sample containers and preservation;
- Receipt within holding times unless qualifying data is acceptable under certain circumstances;
- Adequate sample volume;
- Procedures that are used when samples show signs of damage or contamination; and
- Storage of samples and proper disposal.

5.3 Sample Receipt Protocols

Upon receipt, the condition of the sample is recorded in the comment area of the LIMS if there are any abnormalities or departures from standard condition. All samples which require thermal preservation are considered acceptable if they arrive with ice, cold blue icepacks, or arrival temperature is either within +/-2 degrees C of the required temperature or the method specified range. For samples with a specified temperature of 4°C, samples with a temperature ranging from just above freezing temperature of water to 6°C are considered acceptable. Samples that are hand delivered to the laboratory immediately after collection may not meet this criteria. In these cases, the samples will be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

Where applicable, the MDEQ Field staff document preservation use on the sample bottle label.

Where there is any doubt as to the sample's suitability for testing, the Laboratory may consult its clients for further instruction before proceeding, or may proceed, but will qualify or provide comment on the data when reported. If the sample does not meet the sample receipt acceptance criteria, the Laboratory notes the condition of the samples on the chain of custody form and in the final report and/or in the LIMS.

5.4 Storage Conditions

Samples that require thermal preservation are stored under refrigeration which is +/-2°C of the specified preservation temperature. For samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C is considered acceptable. The main sample storage area is continuously monitored with an emergency notification of key personnel if the temperature exceeds the warning limits. Samples are stored in a manner that prevents cross contamination. For example, standards are stored separately from samples, and volatile samples are stored separately from all other samples.

5.5 Chain of Custody

The Sample Analysis Request Sheet and the Request for Water Analysis are the forms that document the sample chain-of-custody from the field to the laboratory, or from the laboratory to another location as required. A MDEQ Sample Transmittal form will be used for all Enforcement samples submitted to or through the laboratory. The main lab building is a secure and controlled access facility restricted to authorized personnel only. Access is allowed only through coded ID cards issued to authorized staff. In addition, cameras and monitors located throughout the building provide additional security monitoring capabilities.

Once samples are received in the secure environment of the laboratory, the LIMS will provide the necessary documentation of laboratory staff coming in contact with the sample. This LIMS documentation will identify staff, the date of extraction/digestion, and the date of analysis sufficient to reconstruct the sample COC as necessary. Refer to SOP 109, Chain of Custody Procedure for Criminal Court Cases, for further details.

5.6 Sample Disposal

All samples, digestates, leachates and extracts or other sample preparation products are disposed of in accordance with Federal and State laws and regulations. If the sample is part of litigation, disposal of the physical sample occurs only with the concurrence of the affected legal authority, sample data user and/or submitter of the sample. (See SOPs 109, 111, & 119).

6.0 CALIBRATION PROCEDURES AND FREQUENCY

6.1 Traceability of Calibration

Wherever applicable, calibration of analytical support equipment and instruments is traceable to national standards of measurement. Certificates of traceability
are maintained on file and all data can be traced back to these certificates via tracking numbers.

6.2 Reference Standards

Reference standards of measurement (such as Class S or equivalent weights or traceable thermometers) are used for calibration only. Reference standards are subjected to in-service checks between calibrations and verifications.

6.3 General Requirements

Each calibration is dated and labeled with or traceable to the method, instrument, analysis date, and each analyte name, concentration and response (or response factor). Sufficient information is recorded to permit reconstruction of the calibration. Acceptance criteria for calibrations comply with method requirements or are established and documented.

6.4 Analytical Support Equipment

Analytical support equipment includes: autoclaves, balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All such support equipment is:

- maintained in proper working order. The records of all activities including service calls are kept.
- calibrated or verified at least annually, using NIST traceable references when available, at the point of use or over the entire range of use. The results of such calibration must be within the specifications required of the application for which the equipment is used or the equipment is removed from service until repaired.

Balances, ovens, refrigerators, freezers, incubators and water baths are checked with NIST traceable references (where possible) in the expected use range. The acceptability for use or continued use is according to the needs of the analysis or application for which the equipment is being used. Mechanical volumetric dispensing devices used for quantitative purposes (except Class A glassware) are checked each day of use.

6.5 Instrument Calibration

Calibration procedures for a specific laboratory instrument will consist of an initial calibration, or a calibration verification when an initial calibration is not performed on the day of analysis unless otherwise specified in the Laboratory SOP and reference method. The SOP for each

analysis performed in the laboratory describes the calibration procedures, their frequency, acceptance criteria and the conditions that will require recalibration. In all cases, the initial calibration is verified using an independently prepared calibration verification solution. The laboratory maintains a document/instrument logbook for each instrument which contains the following information: instrument identification, serial number, maintenance and may contain the date of calibration, analyst, calibration solutions run and the samples associated with these calibrations.

All results are calculated based on the response curve from the initial calibration and are bracketed by calibration standards or reported as qualified data unless otherwise specified by the SOP (i.e. GC/MS).

If the initial calibration fails, the analysis procedure is stopped and evaluated. For example, a second standard may be analyzed and evaluated or a new initial calibration curve may be established and verified. In all cases, the initial calibration must be acceptable before analyzing any samples.

When an initial calibration is not performed on the day of analysis, a calibration verification check standard is analyzed at the beginning and end of each analytical batch or sequence as specified in the SOP. The concentration of this calibration check varies as described in each method SOP. If a calibration check standard fails, and routine corrective action procedures fail to produce a second consecutive calibration check within acceptance criteria, a new initial calibration curve is constructed. If the continuing calibration acceptance criteria are exceeded high (i.e., high bias), and there are non-detects for the corresponding analyte in all samples associated with the continuing calibration check, then those non-detects may be reported, otherwise the samples affected by the unacceptable check are reanalyzed after a new calibration curve has been established, evaluated and accepted. If the continuing calibration acceptance criteria are below the acceptance limit, results may be reported if sample results indicate a concentration above an action level and/or accurate values are not required by the client. Data will be flagged accordingly. Otherwise, additional sample analysis does not occur until a new calibration curve is established and verified. See analytical SOP's for specific details.

7.0 TEST METHODS AND STANDARD OPERATING PROCEDURES

The Laboratory maintains Standard Operating Procedures (SOPs) that accurately reflect all laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods. Copies of all SOPs are accessible to all staff from the immediate supervisor or via the Intranet. Each SOP indicates the effective date, the revision number, and the signature(s) of the Analyst, QA Officer, Technical Director, Compliance Officer, and Laboratory Director. The electronic copy on the server is considered to be the sole controlled document. All other copies or printouts are considered to be uncontrolled copies. All laboratory personnel are provided with computers to access the controlled document, but do not have the security rights to modify the existing controlled electronic document.

SOPs are used to ensure consistency and to save time and effort. Any deviation from an established procedure during an analysis is documented.

7.1 SOPs for Sample Management (SOP's 105, 109, 117, 119, 121, 122, 124)

The Sample Management SOPs describe the receipt, handling, scheduling, and storage of samples.

Sample Receipt and Handling -- These procedures describe the precautions to be used in opening sample shipment containers and how to verify that chain-of-custody has been maintained, examine samples for damage, check for proper preservatives and temperature, and log samples into the laboratory sample system.

Sample Scheduling -- These procedures describe the sample scheduling in the laboratory and include procedures used to ensure that holding time requirements are met.

Sample Storage -- These procedures describe the storage conditions for all samples, verification and documentation of daily storage conditions, and how to ensure that custody of the samples is maintained while in the laboratory.

7.2 Reagent/Standard Preparation

The method SOPs describe how to prepare specific standards, reagents, selection of specific grades of materials used in reagent and standard preparation, use of appropriate glassware and containers for preparation and storage, and proper labeling. Use of Stock Tracking Logbooks will maintain the documentation and recordkeeping for traceability. The following process will be used to complete the Stock Tracking Logbooks:

7.2.1 Stock Tracking Logbooks

1. All chemicals, solvents, acids, bases, reagents and standards received must be logged into the Stock Tracking Logbook.

2. Any of the above items that go directly to the Filley Street facility will have a receipt date

and a Filley number assigned with a prefix "FL-" and will be logged into a special Filley Street Logbook located at Filley Street.

3. Tracking logbooks will be bound into color-coded books.

4. The Environmental Lab will have a prefix of "EL-" and the Drinking Water Lab will have a prefix of "DW"assigned to all items in the Stocking Tracking Logbook and a receipt date will be documented for tracking purposes.

5. Any items arriving from Filley Street to building #44 must have a "FL" number on the container and must be logged into the Stock Tracking Logbook.

6. All certificates of analysis must have the "EL" or "DW" number written on the top right hand corner by the receiver. The original certificate is to be given to the Laboratory Compliance Officer or the QA Officer and a copy is to accompany the item into the lab.

7. Every container must have a tracking number written on it. (i.e. if a box of 4 bottles is received, these items must be written on each bottle.)

8. When a bottle is opened the opened date and initials of the person opening the bottle must be written on the bottle.

9. If multiple bottles of the same item are received at the same time with the same lot number, one line in the Stock Tracking Logbook may be filled out with a dash and then 1 through X (X represents the number of bottles.) (example: Received a box of methanol containing 4 bottles with all the same lot numbers. Let's assume the next entry in the Stock Tracking Logbook will be "EL-1020". The entry in the book for these bottles of methanol will be "EL-1020-1 through 4.". NOTE: Each bottle will have a unique number written on it. The numbers would be EL-1020-1; EL-1020-2; EL-1020-3; and EL-1020-4.

10. If the item was ordered by credit card put "Charge" in the PO # column.

11. If the item was received from Filley Street and the PO# and vendor are not easily found they can be found on the packing slip which Procurement should have.

7.3 General Laboratory Techniques

SOPs may be developed to describe all essentials of laboratory operations that are not addressed elsewhere. These techniques should include, but are not limited to, safety, sample submission, and chain-of-custody.

7.4 SOPs for Test Methods

Procedures for test methods describing how the analyses are actually performed in the laboratory are specified in method SOPs. These SOPs for sample preparation, cleanup and analysis are

based on reference methods published by EPA, ASTM, AWWA, and other organizations and on internally developed methods validated according to EPA's Performance-Based Measurement System. See SOP 108, Methodology Write Up Format . Each method SOP may include:

1) Applicable analytical methods;
2) applicable matrix or matrices;
3) method detection limits and Reporting Limits;
4) scope and application, including components to be analyzed;
5) method summary;
6) definitions;
7) interference's;
8) safety;
9) equipment and supplies;
10) reagents and standards;
11) sample collection, preservation, shipment and storage;
12) quality control;
13) calibration and standardization;
14) procedure;
15) calculations;
16) method performance;
17) pollution prevention;
18) data assessment;
19) corrective actions;
20) waste management;
21) references; and
22) any tables, diagrams, flowcharts and validation data
23) attachments

7.5 Equipment Calibration and Maintenance

SOPs may be developed to describe how to ensure that laboratory equipment and instrumentation are in working order if it is not addressed in the Method SOP. These procedures may include calibration procedures and schedules, maintenance procedures and schedules, maintenance logs, service arrangements for all equipment, and spare parts available in-house. Calibration and maintenance of laboratory equipment and instrumentation are in accordance with manufacturers' specifications or applicable test specifications.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 Laboratory Quality Control Samples

The data acquired from QC procedures are used to determine the quality of analytical data, and the need for corrective action in response to identified deficiencies, and to interpret results after corrective action procedures are implemented. Each method SOP includes a QC section that addresses the minimum QC requirements for the procedure. The internal QC checks may differ slightly for each individual procedure but in general are described below. The acceptance limits and corrective actions for these QC checks are described in Section 12 and 13 of this Manual.

a) Method Blanks/Laboratory Reagent Blanks are performed at a minimum frequency of one per batch of samples per matrix type per sample extraction or preparation test method. The results of these samples are used to determine possible contamination of the test system.

b) LCS/LFB (QC Check Sample) are analyzed at a minimum of 1 per batch of 20 or fewer samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as (but not limited to) total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples are used to determine batch acceptance (see also MS/MSD for organic testing).

c) Matrix Spikes (MS) are performed at a minimum frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as (but not limited to), total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The sample(s) selected for spiking are rotated among received samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike generally indicates a problem with the sample composition, and not the laboratory analysis, and is reported to assist in data assessment.

d) Surrogates - Surrogate compounds are added to all samples, standards, and blanks for all organic chromatography test methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery generally indicates a problem with the sample composition and is reported to assist in data assessment.

e) Spike Components - In general, the spike compounds are those specified in the method as the spiking components or as the recommended spiking components (such methods 8270 or 625). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs), the test method has an extremely long list of components (such as Methods 8270 or 6010) or components are incompatible, a representative number (10%) of the listed components may be used. The components of each spiking mix represent all chemistries, elution patterns and masses and include permit-specified analytes and other client requested components. All reported components are used in the spike mixture within a two-year time period, and no one component or components dominates the spike mixture.

f) Matrix Spike Duplicates (MSDs) or Laboratory Duplicates are analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation test method. The selected sample(s) are rotated among received samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates generally indicates a problem with the sample composition and is reported to assist in data assessment.

8.2 Method Detection Limits (MDL) and Reporting Limits (RL)

For analytes for which spiking is a viable option, Method Detection Limits are determined as described in 40 CFR Part 136, Appendix B. The method detection limit is initially determined for the compounds of interest in each method in laboratory pure reagent water or sand.

An MDL study is not performed for any component for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature dissolved oxygen or turbidity. For these types of analytes, the reporting limit is based on the signal to noise ratio, from the analysis of a QC check sample, or calibration standard.

The MDEQ Laboratory recognizes that it is impractical to routinely report to the MDL for actual samples. Based on the calculated MDL, a Reporting Limit (RL) is determined, using the specific reference method, the MDL spiking concentration, or a concentration based on these factors and Best Professional Judgement. For this reason, the RL is used as the routine quantitation limit. Typically, it may be at or near the spike concentration used to determine the MDL. In addition, for ease and convenience of reporting, the RL may be: (1) rounded up to the nearest one, two, five, or ten; or (2) rounded to a uniform number for a group of compounds in a gas chromatograph scan. In any case, the RL must be at or above the MDL and have a calibration standard at or below the RL. The reference SOP may take precedence in the selection of the low standard. See Admin SOP 103, "Method Detection Limit (MDL) and Reporting Limit (RL)". Sample results between the RL and the MDL may be reported to the client with a qualification flag indicating a lower confidence in the quantification process.

8.3 Selectivity

a) Absolute and relative retention times aid in the identification of components in chromatographic analyses and help evaluate the effectiveness of a column to separate constituents. Acceptance criteria for retention time windows are documented in each method SOP.

b) A confirmation may be performed to verify a compound identification when positive results are detected on a sample from a location that has not been previously tested. Such confirmations are performed on organic tests except when the analysis involves the use of a mass spectrometer. Confirmation will be performed on a separate column, retesting, or by a different method.

c) Acceptance criteria for mass spectral tuning are contained in SOPs.

8.4 Initial Demonstration of Capability (IDOC)

Prior to acceptance and use of any method, satisfactory initial demonstration of method performance is required. This initial demonstration of method performance is performed each time there is a significant change in instrument type, personnel or test method. An IDOC is completed for each analyst documenting that this activity has been performed. The Unit Manager submits the IDOC to the QA Officer. The IDOC procedure is outlined in Appendix A.

9.0 DATA REDUCTION, REVIEW, REPORTING AND RECORDS

9.1 Data Reduction and Review

Analytical data is reduced according to protocols described in the laboratory SOPs. Computer programs used for data reduction are validated before use by manual calculations. All information used in the calculations (e.g., raw data, calibration files, tuning records, results of standard additions, interference check results, sample response, and blank or background-correction protocols) are recorded in order to enable reconstruction of the final result at a later date. Information on the preparation of the sample (e.g., weight or volume of sample used, percent dry weight for solids, extract volume, dilution factor used) is maintained in order to enable reconstruction of the final result at a later date.

Data may be entered into the LIMS after peer review or at the completion of the originating analyst review and approval. A draft of data entered into the LIMS is reviewed by a second analyst or supervisor according to laboratory procedures to ensure that the QC is acceptable, calculations are correct, qualifications are noted as required, and to detect transcription errors. Spot checks are performed on computer calculations to verify program validity. Errors detected in the review process are referred to the analyst(s) for corrective action. Once the data is validated, the report is finalized and printed for distribution.

As described in Section 12.0, the results of all quality control sample analyses are reviewed and evaluated before data are reported.

9.2 Report Format and Contents

The results of each test, or series of tests are normally reported in a report and include all the information necessary for the interpretation of the results. Each report typically includes:

1) name and address of laboratory, and phone number with name of contact person;
2) a unique identification number and the total number of pages, with all pages sequentially numbered;
3) name and address of client;
4) description and unambiguous identification of the sample(s) including the client identification code;
5) identification of results for any sample that did not meet sample acceptance requirements;
6) date of sample collection and date of sample receipt;
7) identification of the test method used;
8) if the laboratory collected the sample, reference to sampling procedure;
9) any deviations from, additions to or exclusions from SOPs, and any conditions that may have affected the quality of results, and including the use and definitions of data qualifiers;
10) measurements, examinations and derived results, supported by tables, graphs, sketches and photographs as appropriate, and any failures identified; identification of whether data are calculated on a dry weight or wet weight basis; identification of the reporting units such as ug/L or mg/Kg;

11) clear identification of all test data provided by outside sources, such as subcontracted laboratories, clients, etc.; and
12) clear identification of numerical results.

Exceptions to this standard approach for reporting are allowed with approval of the Technical Director and are documented.

Material amendments to a test report after issue are made only in the form of a further document, or data transfer including the statement "Supplement to Test Report, identification number." If the test report is regenerated with corrections, the new report will be clearly marked as "CORRECTED COPY" and both copies, original and corrected, will be filed and maintained as part of the official laboratory documents. For the Drinking Water LABLIMS program, the new report will be clearly marked as "CORRECTED REPORT". Subsequent reports can be generated from LABLIMS at any time using the saved data files

Clients are notified promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a report.

9.3 Records

Records provide the direct evidence and support for the necessary technical interpretations, judgments, and discussions concerning laboratory results. These records, particularly those that are anticipated to be used as evidentiary data, provide the historical evidence needed for later reviews and analyses. Records should be legible, identifiable, and retrievable, and protected against damage, deterioration, or loss. All records referenced in this section are retained for a minimum of seven years in accordance with the departments Record Retention Schedule.

Laboratory records generally consist of bound notebooks with pre-numbered pages, personnel qualification and training forms, equipment maintenance and calibration forms, chain-of-custody forms, sample analysis request forms, and analytical change request forms. All records are recorded in indelible ink and retained for seven years. Records that are stored or generated by computers or personal computers (PCs) have hard copy or write-protected backup copies.

Any documentation errors are corrected by drawing a single line through the error so that it remains legible and is initialed by the responsible individual, along with the date of change. The correction is written adjacent to the error. The person who performed the instrumental analysis signs strip-chart recorder printouts. If corrections need to be made in computerized data, a system parallel to the corrections for handwritten data is used.

Laboratory records include the following:

Standard Operating Procedures -- Any revisions to laboratory procedures are written, dated, and distributed to all affected individuals to ensure implementation of changes. These are readily available to all staff on the laboratory Intranet. An archive of obsolete SOPs are maintained to

determine how historical data was derived. Each revision is given an effective date and an obsolete date in order to define the time frame that the SOP was used.

Equipment Maintenance Documentation -- Documents detailing the receipt and specification of analytical equipment is retained. A history of the maintenance record of each system serves as an indication of the adequacy of maintenance schedules and parts inventory. As appropriate, the maintenance guidelines of the equipment manufacturer are followed. When maintenance is necessary, it is documented in either standard forms or in logbooks.

Calibration Records & Traceability of Standards/Reagents -- The frequency, conditions, standards, and records reflecting the calibration history of a measurement system are recorded.

Sample Management -- A record of all procedures to which a sample is subjected while in the possession of the laboratory is maintained. These include records pertaining to:
a) Sample preservation including appropriateness of sample container and compliance with holding time requirement;
b) Sample identification, receipt, acceptance or rejection and log-in;
c) Sample storage, transmittal forms, and internal routing and assignment records;
d) Disposal of hazardous samples including the date of sample or subsample disposal and name of the responsible person;
e) Automated sample-handling systems.

Original Data -- The raw data and calculated results for all samples is maintained in laboratory notebooks, logs, benchsheets, files or other sample tracking or data entry forms. Instrumental output is stored in a computer file or a hard copy report. These records include:
a) Laboratory sample ID code;
b) Date of analysis;
c) Instrumentation identification and instrument operating conditions/parameters;
d) Analysis type and sample preparation information, including sample aliquots processed, cleanup, and separation protocols.;
e) All manual, automated, or statistical calculations;
f) Confirmatory analysis data, when required to be performed; and
g) Analyst's or operator's initials/signature.

QC Data -- The raw data and calculated results for all QC samples and standards are maintained in the manner described in the preceding paragraph. Documentation allows correlation of sample results with associated QC data. Documentation also includes the source and lot numbers of standards for traceability. QC samples include, but are not limited to, control samples, method blanks, matrix spikes, and matrix spike duplicates.

Deviations -- All deviations from SOPs are reviewed and approved by the QA Officer or Technical Director.

Final Report -- A copy of any report issued and any supporting documentation.

Administrative Records -- The following are maintained:
a) Personnel qualifications, experience and training records;
b) Initial demonstration of proficiency for each analyst; and
c) A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record.

9.4 Document Control System

A document control system is used to ensure that all staff have access to current policies and procedures at all times. Documents which are managed by this system include this Quality Manual and all SOPs. The system consists of a document review, revision and approval system, and, document control and distribution.

All quality documents (this Manual, SOPs, policies, etc.) are reviewed and approved by the QA Officer, Compliance Officer, Technical Director and the Laboratory Director. Such documents are revised whenever the activity described changes significantly. All documents are reviewed at least every 3 years.

The Compliance Officer controls all quality documents. Controlled copies are available \ in read-only .pdf format only on the DEQ Intranet at P:\ESSD\LSS\SOP Directory\Drinking Water SOP's. Uncontrolled copies may be printed as needed by staff, however, the controlled document remains on the website.

9.5 Confidentiality

All laboratory results and associated raw data are kept in confidence to the client who requested the analyses. Freedom of Information Act (FOIA) requests shall follow all rules and regulations as specified by the department and division. Access to laboratory records and LIMs data is limited to laboratory personnel except with the permission of the Unit Managers, Compliance Officer or Laboratory Director. Laboratory & personnel records may be made available to authorized accrediting authority personnel.

Where clients require transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, staff will ensure confidentiality is preserved.

10.0 PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

10.1 Internal Laboratory Audits

Annual internal audits are performed to verify that laboratory operations continue to comply with the requirements of the quality system. The quality assurance officer does such audits. Upon completion of the audit, the QA Officer generates an inspection report citing any deficiencies discovered during the audit. The report is given to the Laboratory Director with a copy to the appropriate Unit Manager and Compliance Officer. It is the ultimate responsibility of the Laboratory Director to ensure that proper corrective action has been implemented on a permanent basis. Where the audit findings cast doubt on the correctness or validity of the laboratory's results, an immediate corrective action is initiated and any client whose work may have been affected is notified.

The internal system audits include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc.

10.2 Managerial Review

At least once per year, laboratory management conducts a review of the quality system to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The review takes account of reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions and other relevant factors.

11.0 FACILITIES, EQUIPMENT, REAGENTS, AND PREVENTATIVE MAINTENANCE

11.1 Equipment

Records are maintained for each major equipment and all reference materials significant to the tests performed. These records include documentation on all routine and non-routine maintenance activities and reference material verifications.
The records include:
1) the name of the equipment;
2) the manufacturer's name, type identification, and serial number or other unique identification;
3) date received and date placed in service (if available);
4) current location, where appropriate;
5) if available, condition when received (e.g. new, used, reconditioned);
6) copy of the manufacturer's instructions, where available;
7) dates and results of calibrations;
8) details of maintenance carried out to date and planned for the future; and
9) history of any damage, malfunctions, modification or repair.

Glassware Cleaning - Glassware is cleaned to meet the requirements of the method. Any cleaning and storage procedures that are not specified by the method are documented in laboratory records and SOPs.

11.2 Documentation and Labeling of Standards and Reagents

Records are kept for all standards, including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if supplied), the date of receipt, recommended storage conditions, and an expiration date after which the material is not used unless it is verified. See also Section 7.2.

Original containers provided by the vendor are labeled with an expiration date.

Detailed records are maintained on reagent and standard preparation. These records indicate traceability to purchased stocks or neat compounds, details of preparation, date of preparation, expiration date and preparer's initials.

All containers of prepared reagents and standards bear a unique identifier and expiration date and are linked to the documentation requirements above.

1) Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the method are not to be used. The labels on the container are checked to verify that the purity of the reagents meets the requirements of the particular method. Such information is documented.

2) Water - The quality of reagent water sources is monitored and documented to meet method specified requirements.

11.3 Computers and Electronic Data Related Requirements

Where computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data:

- Computer software is documented to be adequate for use (provided by vendor);
- Procedures are established and implemented for protecting the integrity of data;
- Computer and automated equipment are maintained to ensure proper functioning;
- Appropriate procedures are used for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records through password protection and read only attributes.

11.4 Preventative Maintenance

A routine preventative maintenance program is used to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees regularly perform routine scheduled maintenance and repair of all instruments. All maintenance that is performed is documented in the instrument maintenance logbook. All laboratory instruments are maintained in accordance with manufacturer's specifications. Maintenance will be specified by the manufacturer, reference method and in the SOPs.

11.5 Inspection/Acceptance Requirements for Supplies and Consumables

Labels indicating the following information on receipt and testing are to be used for critical supplies and consumables.

- Unique identification number (if not clearly shown).
- Date received and initials.
- Date opened and initials.
- Expiration date.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO EVALUATE DATA QUALITY

Quality control acceptance criteria are used to determine the validity of the data based on the analysis of internal quality control check (QC) samples (see Section 8.0). The specific QC samples and acceptance criteria are found in the laboratory SOPs. Typically, laboratory established acceptance criteria are taken from published EPA methods. Where no EPA criteria exist, laboratory generated acceptance criteria are established. Acceptance criteria for bias are based on the historical mean recovery plus or minus three standard deviations from the mean, and acceptance criteria for precision range from zero (no difference between duplicate control samples) to the historical mean relative percent difference plus three standard deviation units.

Analytical data generated with QC samples that fall within prescribed acceptance criteria indicate the laboratory was in control. Data generated with QC samples that fall outside the established acceptance criteria indicate the laboratory was "out-of-control" for the failing tests. These data are considered suspect and the corresponding samples are reanalyzed or reported with qualifiers.

Many published EPA methods do not contain recommended acceptance criteria for QC sample results. In these situations, the MDEQ Environmental Laboratory uses 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house limits are developed. In-house limits are based on a 95% confidence interval and must include a minimum of 20 data points.

12.1 Laboratory Control Samples or Fortified Blanks

A laboratory control sample (LCS) or fortified blank (LFB) is analyzed with each batch of samples to verify that the accuracy of the analytical process is within the expected performance of the method. The results of the laboratory control sample are compared to acceptance criteria to determine usability of the data. Data generated with LCS/LFB samples that fall outside the established acceptance criteria are judged to be out-of-control. These data are considered suspect and the corresponding samples are reanalyzed or reported with qualifiers. See specific Organic SOP's for use of MS/MSD vs LCS/LCSD.

12.2 Matrix Spikes/Matrix Spike Duplicates

Results from MS/MSD analyses are primarily designed to assess data quality in a given matrix, and not laboratory performance. In general, if the LCS results are within acceptance criteria, performance problems with MS/MSD results may either be related to the specific sample matrix or to an inappropriate choice of extraction, cleanup, or determinative methods. If any individual percent recovery in the matrix spike (or matrix spike duplicate) falls outside the designated acceptance criteria, the laboratory will determine if the poor recovery is related to a matrix effect or a laboratory performance problem. A matrix effect is indicated if the LCS data are within acceptance criteria but the matrix spike data exceed the acceptance criteria.

12.3 Surrogate Recoveries

Surrogates are used in organic analyses. Surrogate recovery data from individual samples are compared to laboratory generated limits (and reference surrogate recovery acceptance criteria if available). As for MS/MSD results, surrogate recoveries are used primarily to evaluate data quality and not laboratory performance.

12.4 Method Blanks

Method blank analyses are used to assess acceptance of sample results and to demonstrate the presence of any interferences or contamination throughout the entire analytical process, including sample preparation. The source of contamination is investigated and measures taken to correct, minimize or eliminate the problem if:

- the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch or
- the blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.
- any blank value above the RL is reported with the appropriate qualifying remark code

Each sample in the affected batch is assessed against the above criteria to determine if the sample results are acceptable. Any sample associated with the contaminated blank is reprocessed for analysis or the result is reported with appropriate data qualifying codes.

13.0 CORRECTIVE ACTION

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out of control QC performance that can affect data quality. To the extent possible, samples are reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure are reported with the appropriate data qualifier(s). Sample results may also be qualified when holding times are not met, improper sample containers and/or preservatives are used or when other deviations from laboratory standard practices and procedures occur.

Corrective action in the laboratory may occur prior to, during and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, potentially high concentration samples may be identified during sample log-in or just prior to analysis. The SOPs specify conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, and automatic reinjection/reanalysis when certain QC criteria are not met.

Any QC sample result outside of acceptance limits requires corrective action. Once the problem has been identified and addressed, corrective action may include the reanalysis of samples, or appropriately qualifying the results.

The analyst will identify the need for corrective action. The Unit Technical Director will approve the required corrective action to be implemented by the laboratory staff. The QA Officer will ensure implementation and documentation of the corrective action.

Corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both a corrective action log (signed by analyst, Unit Technical Director, and QA Officer), and the narrative in data report. Reported results that have been qualified do not require entry into the corrective action log.

Where a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the quality of the laboratory's tests, the laboratory shall ensure that those areas of activity and responsibility involved are promptly audited. Records of the complaint and subsequent actions are maintained.

14 SUBCONTRACTING AND SUPPORT SERVICES AND SUPPLIES

14.1 Contracting Laboratory Services

- The MDEQ Environmental Laboratory has eight State Contract Laboratories available as overflow labs. Field staff submitting samples to the DEQ Laboratory will designate the appropriate contract laboratory for referral if such funding is available. Clients are advised if any analyses will be referred to a contract laboratory. At this time, all reporting and invoicing transactions for samples referred to a contract laboratory will be handled between the client and the contracting laboratory.
- The State Contract Laboratories must have an MDEQ approved QAPP, are subject to on-site audits, and may have their data audited to ensure acceptable quality.

14.2 Outside Support Services and Supplies

The Laboratory only uses those outside support services and supplies that are of adequate quality to sustain confidence in the laboratory's tests. Records of all suppliers for support services or supplies required for tests are maintained.

15.0 REFERENCES

NELAC Standards, July 1999

Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80, December 29, 1980, Office of Monitoring Systems and Quality Assurance, ORD, U.S. EPA, Washington, DC 20460.

RCRA QAPP Instructions, U. S. EPA Region 5, Revision: April 1998

ASTM D-5283-92.Generation of Environmental Data Related to Waste Management Activities: Quality assurance and Quality Control Planning and Implementation

"American National Standards Specification and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs (ANSI/ASQC E-4)", 1994

EPA 2185 - Good Automated Laboratory Practices, 1995

ISO/IEC Guide 25: 1990. General requirements for the competence of calibration and testing laboratories

QA/R-2: EPA Requirements for Quality Management Plans, August, 1994

QA/G-4: Guidance for the Data Quality Objectives Process EPA/600/R-96/055, September, 1994

QA/R-5: EPA Requirements for Quality Assurance Project Plans Draft - November 1997

QA/G-5: Guidance on Quality Assurance Project Plans EPA/600/R-98/018, February, 1998

QA/G-6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November, 1995

QA/G-9: Guidance for the Data Quality Assessment: Practical Methods for Data Analysis EPA/600/R-96/084 , January, 1998

Manual for the Certification of Laboratories Analyzing Drinking Water EPA/570/9-90/008

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

LABORATORY SERVICES SECTION
ENVIRONMENTAL LABORATORY
QUALITY ASSURANCE PROGRAM PLAN

APPENDIX A

Initial Demonstration of Capability

QAPP-Ver2.0, Revison 0
May 5, 2003

APPENDIX A
INITIAL DEMONSTRATION OF CAPABILITY (IDOC)

A demonstration of capability (DOC) is made prior to using any test method, and at any time there is a significant change in instrument type, personnel or test method.

All demonstrations are documented through the use of the form in this appendix.

The following steps are performed.

a) A quality control sample is obtained from an outside source. If not available, the QC sample is prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The analyte(s) are diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration approximately 10 times the laboratory-calculated detection limit.

c) Four aliquots are prepared and analyzed according to the test method either concurrently or over a period of days.

d) Using all of the results, the mean recovery and the standard deviation is calculated for each parameter of interest.

e) The calculated mean and standard deviation are compared to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

f) When one or more of the tested parameters fail at least one of the acceptance criteria, the laboratory repeats the test for all parameters that failed to meet criteria. If repeated failure occurs, the laboratory will locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

g) For those analysts that have past recognized historical laboratory analytical training, experience, responsibilities and duties, the use of recent control data may be used for the purposes of documenting an IDOC.

Documentation

The IDOC data is used to document the completion of each demonstration of capability. The Unit Manager will review the data, accept/reject, and forward to the QA Officer. A copy will be maintained by the QA Officer, analyst, and staff record file.

Michigan Department of Environmental Quality
Laboratory Section
Drinking Water and Radiological Protection Division
3350 N. Martin Luther King Jr. Blvd.
Lansing MI, 48909-0777

DATE:
ANALYST:
METHOD:
MATRIX:

SUPERVISOR APPROVAL:

INITIAL DEMONSTRATION OF CAPABILITY

Analyte	Spike Value (units)	REP #1	REP #2	REP #3	REP #4	STANDARD DEVIATION	MEAN	%RSD	Acceptance Criteria	RESULTS ACCEPTABLE?	COMMENTS
Example		7.00	4.00	5.00	4.00	1.41	5.00	28.28	20.00	Fail	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	
Example		5.00	4.00	5.00	4.00	0.58	4.50	12.83	20.00	Pass	

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

LABORATORY SERVICES SECTION
ENVIRONMENTAL LABORATORY
QUALITY ASSURANCE PROGRAM PLAN

APPENDIX B

List of Standard Operating Procedures (SOP's)

QAPP-Ver2.0, Revison 0
May 5, 2003

APPENDIX B

TABLE OF CONTENTS - STANDARD OPERATION PROCEDURE (SOP)
DWRPD LABORATORY SERVICES SECTION

UNIT	SOP #	TYPE	TITLE
Administrative	SOP 100	N/A	Safety
Administrative	SOP 101	N/A	Minimal Requirements for an Analytical Program
Administrative	SOP 102	N/A	Requisition of Supplies
Administrative	SOP 103	N/A	Method Detection Limit (MDL) and Reporting Limit (RL)
Administrative	SOP 104	N/A	Laboratory Safety Inspections
Administrative	SOP 105	N/A	Lab Opening and Closing
Administrative	SOP 106	N/A	Lab Result Remark Codes
Administrative	SOP 107	N/A	Methodology Approval
Administrative	SOP 108	N/A	Methodology Write-Up Format
Administrative	SOP 109	N/A	Chain of Custody for Criminal Court Cases
Administrative	SOP 110	N/A	Field Blanks and Field Replicates
Administrative	SOP 111	N/A	Handling of Laboratory Wastes and Materials
Administrative	SOP 112	N/A	Handling of “A” Designated Carcinogens
Administrative	SOP 113	N/A	Emergency Evacuation
Administrative	SOP 114	N/A	Equipment Procurement
Administrative	SOP 115	N/A	Method Development Request
Administrative	SOP 116	N/A	Cost Accounting

UNIT	SOP #	TYPE	TITLE
Administrative	SOP 117	N/A	Identifying Samples Known or Suspected to be Hazardous
Administrative	SOP 118	N/A	Annual Precision and Accuracy Summaries
Administrative	SOP 119	N/A	Storage of Samples and Lab Data
Administrative	SOP 120	N/A	Recycling
Administrative	SOP 121	N/A	Sampling Techniques and Sample Submittal
Administrative	SOP 122	N/A	Sample Receiving/Drinking Water Laboratory
Administrative	SOP 123	N/A	Notification of Positive Bacteriological Samples (E. coli and Repeat Total Coliform)
Administrative	SOP 124	N/A	Sample Receiving/Environmental Laboratory
Environmental Inorganic	SOP 200	Sample Prep.	ASTM Leachate Procedure
Environmental Inorganic	SOP 201	Sample Prep.	Acid Digestion for Arsenic, Selenium, and Antimony
Environmental Inorganic	SOP 202	Sample Prep.	Extraction Procedure Toxicity (EP Tox.)
Environmental Inorganic	SOP 203	Sample Prep.	Acid Digestion for Metals (Hot Plate)
Environmental Inorganic	SOP 204	Sample Prep.	Solid Digestion for Arsenic and Selenium
Environmental Inorganic	SOP 205	Sample Prep.	Sediment Drying Procedure for Metals
Environmental Inorganic	SOP 206	Sample Prep.	Synthetic Precipitation Leaching Procedure (SPLP)
Environmental Inorganic	SOP 207	Sample Prep.	Microwave Digestion for Metals in Water
Environmental Inorganic	SOP 208	Sample Prep.	Microwave Digestion for Metals in Organic Matrices
Environmental Inorganic	SOP 209	Sample Prep.	Sediment Digestion for Metals
Environmental Inorganic	SOP 210	Sample Prep.	Sediment Digestion for Arsenic
Environmental Inorganic	SOP 211	Sample Prep.	Extraction of Filter Material

UNIT	SOP #	TYPE	TITLE
Environmental Inorganic	SOP 300	Analytical	Chloride (Colorimetric, Automated Ferricyanide, Traacs 800)
Environmental Inorganic	SOP 301	Analytical	Alkalinity (Colorimetric, Automated Methyl Orange, Traacs 800)
Environmental Inorganic	SOP 302	Analytical	Sulfate (Colorimetric, Automated Methylthymol Blue, Traacs)
Environmental Inorganic	SOP 303	Analytical	Determination of Total Kjeldahl Nitrogen in Water
Environmental Inorganic	SOP 304	Analytical	Determination of Total Phosphorus in Water
Environmental Inorganic	SOP 305	Analytical	Biological Oxygen Demand (BOD)
Environmental Inorganic	SOP 306	Analytical	Ultimate Biochemical Oxygen Demand (UBOD)
Environmental Inorganic	SOP 307	Analytical	Chemical Oxygen Demand (COD) - Low Level in Water
Environmental Inorganic	SOP 308	Analytical	Chemical Oxygen Demand (COD) - High Level in Water
Environmental Inorganic	SOP 309	Analytical	Chemical Oxygen Demand (COD) - Total in Sediments
Environmental Inorganic	SOP 310	Analytical	Chlorophyll A
Environmental Inorganic	SOP 311	Analytical	Conductivity
Environmental Inorganic	SOP 312	Analytical	Cyanide, Total
Environmental Inorganic	SOP 313	Analytical	Cyanide, Amenable to Chlorination
Environmental Inorganic	SOP 314	Analytical	Flash Point
Environmental Inorganic	SOP 315	Analytical	Dissolved Oxygen (DO)
Environmental Inorganic	SOP 316	Analytical	Ammonia
Environmental Inorganic	SOP 317	Analytical	Kjeldahl Nitrogen (Total in Solids)
Environmental Inorganic	SOP 318	Analytical	Nitrate Plus Nitrite
Environmental Inorganic	SOP 319	Analytical	Nitrite in Water

UNIT	SOP #	TYPE	TITLE
Environmental Inorganic	SOP 320	Analytical	Orthophosphate in Water
Environmental Inorganic	SOP 321	Analytical	Dissolved Orthophosphate
Environmental Inorganic	SOP 322	Analytical	Percent Total Solids
Environmental Inorganic	SOP 323	Analytical	Percent Volatile Solids
Environmental Inorganic	SOP 324	Analytical	Phenolics
Environmental Inorganic	SOP 325	Analytical	Pheophytin A
Environmental Inorganic	SOP 326	Analytical	Residue – Total Settleable
Environmental Inorganic	SOP 327	Analytical	Residue – Total Dissolved
Environmental Inorganic	SOP 328	Analytical	Residue – Total Solids
Environmental Inorganic	SOP 329	Analytical	Residue – Total Suspended Solids
Environmental Inorganic	SOP 330	Analytical	Residue – Suspended Volatile Solids
Environmental Inorganic	SOP 331	Analytical	Reactive Silicates
Environmental Inorganic	SOP 332	Analytical	Sulfide (Titrimetric in Water)
Environmental Inorganic	SOP 333	Analytical	Sulfide (Total in Water)
Environmental Inorganic	SOP 334	Analytical	Total Organic Compound (TOC)
Environmental Inorganic	SOP 335	Analytical	Total Suspended Particulate (TSP) Hi-Vol Filter
Environmental Inorganic	SOP 336	Analytical	Turbidity
Environmental Inorganic	SOP 337	Analytical	Alkalinity – Phenolphthalein
Environmental Inorganic	SOP 338 (M-6)	Analytical	Arsenic, Selenium, and Antimony by AA-Hydride TO BE UPDATED INTO NELAC FORMAT
Environmental Inorganic	SOP 339	Analytical	Arsenic – Total in Sediments

UNIT	SOP #	TYPE	TITLE
Environmental Inorganic	SOP 340	Analytical	Calcium, Magnesium, Sodium, and Potassium
Environmental Inorganic	SOP 341	Analytical	Fuel Analysis
Environmental Inorganic	SOP 342	Analytical	Hardness (Calculated)
Environmental Inorganic	SOP 343	Analytical	Hexavalent Chromium
Environmental Inorganic	SOP 344 (M-7)	Analytical	Mercury – AA/Cold Vapor (M-7 TO BE UPDATED INTO NELAC FORMAT)
Environmental Inorganic	SOP 345	Analytical	Metals in Hi-Vol Filters
Environmental Inorganic	SOP 346	Analytical	Metals – Total and Dissolved
Environmental Inorganic	SOP 347	Analytical	Metals – AA/Graphite Furnace
Environmental Inorganic	SOP 348 (M-4)	Analytical	Metals – ICP (M-4 TO BE UPDATED INTO NELAC FORMAT)
Environmental Inorganic	SOP 349	Analytical	Metals – ICP/MS
Environmental Inorganic	SOP 350	Analytical	pH
Environmental Inorganic	SOP 351	Analytical	pH – Soil
Environmental Inorganic	SOP 352	Analytical	Selenium – AA/Hydride
Environmental Inorganic	SOP 353	Analytical	Chlorine – Total in Fuels
Environmental Inorganic	SOP 354	Analytical	Gravimetric Determination of Oil and Grease in Water Matrix Samples Via Solid Phase Extraction
Environmental Inorganic	SOP 355	Analytical	Mercury – CVAFS (Gold Trap)
Environmental Inorganic	SOP 356	Analytical	Mercury – CVAFS (Without Gold Trap)
Environmental Organic	SOP 400	Sample Prep.	Gel-Permeation Cleanup (GPC)
Environmental Organic	SOP 401	Sample Prep.	Pressurized Fluid Extraction (Accelerated Solvent Extraction)
Environmental Organic	SOP 402	Sample Prep.	Silica Gel Cleanup

UNIT	SOP #	TYPE	TITLE
Environmental Organic	SOP 403	Sample Prep.	Florisil Cleanup
Environmental Organic	SOP 404	Sample Prep.	Waste Dilution for Semi-Volatile Organic Compounds
Environmental Organic	SOP 405	Sample Prep.	Separatory Funnel Liquid-Liquid Extraction
Environmental Organic	SOP 406	Sample Prep.	Zero Headspace Extraction of Volatile Organic Compounds
Environmental Organic	SOP 500	Analytical	Semi-Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
Environmental Organic	SOP 501	Analytical	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
Environmental Organic	SOP 502 (TO-8)	Analytical	Organochlorine Pesticides, Polychlorinated Biphenyls (PCB's) and Chlorinated Hydrocarbon Organic Compounds by Gas Chromatography with Electron Capture Detection (GC/ECD) (TO-8 TO BE UPDATED INTO NELAC FORMAT)
Environmental Organic	SOP 503	Analytical	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) in Air Canisters
Drinking Water	SOP 600	Microbiology	Heterotrophic Plate Count
Drinking Water	SOP 601	Microbiology	Chromogenic Substrate Coliform Test Using ONPG-MUG (Colilert, Colisure)
Drinking Water	SOP 602	Microbiology	Determination of Fecal Coliforms
Drinking Water	SOP 603	Microbiology	Escherichia Coli and Enterococci in Water by the Membrane Filter Procedure
Drinking Water	SOP 602	Microbiology	Determination of Fecal Coliform by Membrane Filter Procedure
Drinking Water	SOP 603	Microbiology	Escherichia Coli (E. coli) in Water by the Membrane Filter Procedure
Drinking Water	SOP 700 (ARCHIVED)	Inorganics	Determination of Turbidity by Nephelometry
Drinking Water	SOP 700 (REVISION 1)	Inorganics	Determination of Turbidity by Nephelometry
Drinking Water	SOP 701	Inorganics	Automated Testing – Nitrate, Nitrite, Chloride, Fluoride, Sodium, Iron, Hardness, and Sulfate
Drinking Water	SOP 702	Inorganics	Cyanide Analysis by Colorimetric Determination

UNIT	SOP #	TYPE	TITLE
Drinking Water	SOP 703	Inorganics	Determination of Ammonia by Selective Electrode Method
Drinking Water	SOP 704	Inorganics	Conductivity/Resistance of Water by Platinum Electrode
Drinking Water	SOP 705	Inorganics	Alkalinity (Titrimetric, pH 4.5)
Drinking Water	SOP 706	Inorganics	Hydrogen Ion pH Using Glass Electrodes
Drinking Water	SOP 707	Inorganics	Quantitative Hydrogen Sulfide by Titrimetric Determination
Drinking Water	SOP 708	Inorganics	Qualitative Detection of Hydrogen Sulfide
Drinking Water	SOP 709	Inorganics	Qualitative Detection of Tannin-Lignin
Drinking Water	SOP 710	Inorganics	Ortho-Phosphate by Colorimetry
Drinking Water	SOP 711	Inorganics	Fluorescent Dye Testing of Charcoal Packets
Drinking Water	SOP 712	Inorganics	Determination of Silica as SiO_2 by Molybdosilicate Method
Drinking Water	SOP 713	Inorganics	Toluidine Blue Active Substance (TBAS) Screening
Drinking Water	SOP 714	Inorganics	Determination of Perchlorate in Drinking Water Using Ion Chromatography
Drinking Water	SOP 715	Inorganics	Determination of Inorganic Disinfection By-Products in Drinking Water by Ion Chromatography
Drinking Water	SOP 716	Inorganics	Maximum Potential Formation of Total Trihalomethanes
Drinking Water	SOP 717	Inorganics	Analysis of Phenols (Acidic Extract) by GC/FID
Drinking Water	SOP 718	Inorganics	Determination of Ammonia by Selective Electrode
Drinking Water	SOP 719	Inorganics	Conductivity/Resistance of Water by Platinum Electrode
Drinking Water	SOP 720	Inorganics	Alakalinity
Drinking Water	SOP 721	Inorganics	Hydrogen Ion pH Using Glass Electrodes
Drinking Water	SOP 722	Inorganics	Quantitative Hydrogen Sulfide by Titrimetric Determination

UNIT	SOP #	TYPE	TITLE
Drinking Water	SOP 723	Inorganics	Qualitative Detection of Tannin-Lignin
Drinking Water	SOP 800	Metals	Trace Metals Analysis Using ICP/MS
Drinking Water	SOP 801	Metals	Lead Analysis by Zeeman Graphite Furnace Atomic Absorption Spectrometry
Drinking Water	SOP 802	Metals	Metals Analysis by Atomic Absorption Spectrometry
Drinking Water	SOP 803	Metals	Metals Analysis by Flame Photometric Method
Drinking Water	SOP 804	Metals	Metals by Atomic Absorption Spectrometry
Drinking Water	SOP 805	Metals	Potassium
Drinking Water	SOP 806	Metals	Sodium
Drinking Water	SOP 900	Organics	Determination of Endothall in Drinking Water by Ion Exchange Extraction, Acidic Methanol Methylation, and GC/MS
Drinking Water	SOP 901	Organics	Measurement of N-Methyl Carbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Post-Column Derivatization
Drinking Water	SOP 901 (REVISION 1)	Organics	Measurement of N-Methyl Carbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Post-Column Derivatization
Drinking Water	SOP 902	Organics	Analysis of Purgeable Organic Compounds in Drinking Water by Gas Chromatography and ITD Mass Spectrometry
Drinking Water	SOP 903	Organics	Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry
Drinking Water	SOP 904	Organics	Determination of Polynuclear Aromatics and Phthalates in Drinking Water by Solid Phase Extraction and Gas Chromatography and Mass Spectrometry
Drinking Water	SOP 905	Organics	Determination of Diquat and Paraquat in Drinking Water by Solid Phase Extraction and High Performance Liquid Chromatography with Ultra-violet Detection
Drinking Water	SOP 906	Organics	Determination of Chlorinated Acids in Water Using Solid Phase Extraction and GC with an Electron Capture Detector
Drinking Water	SOP 907	Organics	Determination of Haloacetic Acids and Dalapon in Drinking Water by Ion-Exchange Liquid-Solid Extraction and Gas Chromatography with

UNIT	SOP #	TYPE	TITLE
			Electron Capture Detection
Drinking Water	SOP 908	Organics	Determination of Glyphosate in Drinking Water by Direct Aqueous Injection HPLC Post-column Derivatization and Fluorescence Detection
Drinking Water	SOP 909	Organics	Analysis of Purgeable Organic Compounds of Drinking Water by Gas Chromatography and ITD-Mass Spectrometry
Drinking Water	SOP 910	Organics	1,2-Dribromoethane (EDB) and 1,2-Dibromo-3-Chloro Propane (DBCP) in Water by Microextraction and Gas Chromatography
Drinking Water	SOP 911	Organics	Determination of Total Organic Carbon in Drinking Water by UV-Persulfate Oxidation with Nondispersive Infrared Detector (NDIR)
Drinking Water	SOP 912	Organics	Maximum Potential Formation of Total Trihalomethanes
Drinking Water	SOP 913	Organics	Analysis of Phenols
Drinking Water	SOP 914	Organics	Determination of Organophosphate Pesticides in Drinking Water by Solid Phase Extraction and Gas Chromatography/Mass Spectrometry
Drinking Water	SOP 915	Organics	Determination of Pesticides in Drinking Water and Ground Water by High Performance Liquid Chromatography with a Photodiode Array Detector
Drinking Water	SOP 916	Organics	Determination of Pesticides

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

LABORATORY SERVICES SECTION
ENVIRONMENTAL LABORATORY
QUALITY ASSURANCE PROGRAM PLAN

APPENDIX C

Sample Bottle and Preservation Form

QAPP-Ver2.0, Revison 0
May 5, 2003

MDEQ ENVIRONMENTAL LABORATORY

Sample Bottle and Preservative Request Form

Contact : Barry Baker
Phone (517)335-9686
Fax (517)335-9551

Requested by:	*Phone Number:*	*Date(s) Scheduled at MDEQ Laboratory*	TODAY'S DATE / /
Division/District	*Site or Project*	**PLEASE ALLOW AT LEAST 2 WEEKS FOR PREPARATION**	PICK-UP DATE / / AM PM

	Bottle Code	Parameter Group	Plastic Containers: 500 mL White	250 mL White	250 mL Brown	Glass Containers: 40 mL Septum Vial	MeOH Kit*	1000 mL Amber	250 mL (8oz.) Wide	250 mL Narrow	250 mL SC CAP	Preservatives	Need	Prepared
WATER	DO	Dissolved Oxygen										DO KIT ***		
	GN	Gen Chem/Neutral Ortho-phos. Nitrite, BOD, Silica, Residues												
	GA	Gen Chem/Acidic COD, TOC, Ammonia, Kjel N, Tot Phos., Nitrate + Nitrite										H2SO4		
	GG	Gen Chem/ Phenolics-Glass										H2SO4		
	GB	Gen Chem/Basic Cyanide										10 N NaOH		
	S	Sulfide										1 M ZnAC 10 N NaOH		
	CA	Chlorophyll a										1% MgCO3		
	MA	Metals/Total Acidic Cd,Cr,Cu,Ni,Pb,Zn,Fe,Co, Li,Mn,Al,Ti,V,Ba,Be,Tl,Sr, Ag,Hg,As,Se,Sb,Ca,Mg, Na,K										1:1 HNO3		
	MAD	Metals/Acidic Dissolved Parameter List Same as MA, Field Filtered										1:1 HNO3		
	MD	Metals/Dissolved Parameter List Same as MA, Lab Filtered												
	MN	Minerals/Neutral pH, Conduct., Cr+6, Chloride, Sulfate, Alk., Carbonate, Bicarbonate												
	OG	Oil and Grease	OG samples require 2 x 250mL jars per sample									H2SO4		
	VOA	Volatiles 8260/624	VOA requires 2 vials									1:1 HCL		
	ON	PCB/Pesticides 608/8081/8082	ON samples require 2 ambers **											
	BNA	BNA/PNA/BN/Acids 8270/625	BNA samples require 2 ambers **											
SOIL	MS	Metals Parameter List Same as MA												
	GS	Gen Chem/ TP, Kjel-N,Phenolics, COD, Cyanide												
	VOA	Volatiles MeOH/8260*										MeOH		
	OS/BNA	PCB/Pesticides/BNA/PNA/BN 8270/8081/8082												
OIL	MO	Metals/Oil Liq & Solid Waste Parameter List Same as MA												
	OL	Volatiles/PCB/Pesticides/BNA/BN 8260/8270/8081/8082												
	HW	Flash Point/ Solubility												
	MX	Metals/TCLP/SPLP/ASTM for MI-10												
	OX	TCLP/SPLP Volatiles 8260												
		TOTAL REQUESTED												
		NUMBER PREPARED												

Other Requested Supplies:

Please include field QC in the bottle count

MESSAGE RECEIVED BY: _____ PREPARED BY: _____

Refer to back of form for collection and preservation information.

* MeOH Kit includes 1-tared vial, 1-10mL MeOH tube, 1-capped syringe, & 1-syringe label.

** See note #14 on reverse side.

*** DO Kit: DO-1 & DO-2 reagent, must request H2SO4 (unless you already have it)

COLLECTION AND PRESERVATION OF WATER, SEDIMENT, AND OIL SAMPLES

	BOTTLE CODE	PARAMETER GROUP	NOTES	BOTTLE TYPE	SIZE/COMMENTS	COOL TO 4 C° X = YES	PRESERVATIVE	AMOUNT	MAXIMUM HOLDING TIME
WATER	DO	DISSOLVED OXYGEN	1	GLASS	250 mL GLASS STOPPER	X	FIX ON SITE (WINKLER)		8 HR
	GN	GEN CHM/NEUTRAL	2,3	PLASTIC	500 mL	X			2, 7, or 28 DAYS
	GA	GEN CHM/ACIDIC	3,4	PLASTIC	500 mL	X	H2SO4 TO pH < 2	10 DROPS/ 500 mL	28 DAYS
	GG	GEN CHM/PHENOLICS	5	GLASS	250 mL SCREW CAP	X	H2SO4 TO pH<2	5 DROPS/ 250 mL	28 DAYS
	GB	GEN CHM/BASIC	3,6,7,8	PLASTIC	250 mL OR 500 mL	X	10N NaOH TO pH=12	10 DROPS/ 250 mL	14 DAYS (24 HR)
	S	SULFIDE	1	PLASTIC	250 mL	X	2N ZnAC, 10N NaOH TO pH>9	10 DROPS 2 DROPS/ 250 mL	7 DAYS
	CA	CHLOROPHYLL A	9	PLASTIC	250 mL	X	1% MgCO3	5 DROPS/ 250 mL	2 DAYS
	MA	METALS/TOTAL ACIDIC	10	PLASTIC	500 mL		1:1 HNO3 TO pH<2	5 mL/ 500 mL	28 DAYS, 6 MO
	MAD	METALS/ACIDIC FIELD FILTERED	10,11	PLASTIC	500 mL		FILTER ON SITE THEN ADD 1:1 HNO3 TO pH<2	5 mL/ 500mL	28 DAYS, 6 MO
	MD	METALS/LAB FILTERED	11	PLASTIC	500 mL	X	LAB FILT. & PRES.		W/IN 24 HRS.
	MN	MINERAL/NEUTRAL	12	PLASTIC	500 mL	X			W/IN 24 HRS, 2 DAYS, 14 & 28 DAYS
	OG	OIL & GREASE		GLASS	2x250mL W.M.	X	H2SO4 to pH<2	10 DROPS/250mL	28 DAYS
	VOA	VOLATILES 8260/624	13	GLASS	2-40mL SEPTUM VIALS	X	1:1 HCl TO pH<2	5 DROPS/VIAL	14 DAYS
	ON	PCB/PESTICIDES 608/8081/8082	14,17,18	GLASS	2-1000mL AMBER	X			7 DAYS
	BNA	BNA/PNA/BN/ACIDS 625/8270	14,18	GLASS	2-1000mL AMBER	X			7 DAYS
SOIL	MS	METALS		GLASS	250 mL W.M.	X			
	GS	GEN CHEM		GLASS	250mL W.M.	X			
	VOA	VOLATILES MeOH/8260	16	GLASS	MeOH Kit	X	METHANOL	10g/10mL MeOH	14 DAYS
	OS/BNA	PCB/PESTICIDES/BNA/PNA/BN 8270/8081/8082		GLASS	250mL W.M.	X			14 DAYS
OIL	MO	METALS		GLASS	250 mL W.M.	X			
	OL	VOLATILES/PCB/PESTICIDES/BNA/BN	15	GLASS	250 mL W.M.	X			
	HW	FLASH POINT		GLASS	250mL W.M.	X			
	MX	METALS/TCLP/SPLP/ASTM for MI10		GLASS	250mL W.M.	X			
	OX	TCLP/SPLP VOLATILES 8260		GLASS	250 mL SEPTUM JARS	X			14 DAYS

NOTES:

1 EXTRA CARE SHOULD BE TAKEN DURING COLLECTION SO THAT THE SAMPLE IS NOT AERATED BEFORE PRESERVATION.

2 GN, GEN CHM/NEUTRAL, INCLUDES THE FOLLOWING PARAMETERS WITH THEIR HOLDING TIMES: SETTLEABLE RESIDUE, NITRITE, ORTHOPHOSPHATE, BOD, AND TURBIDITY (2 DAYS); TOTAL, FILTERABLE, NONFILTERABLE AND VOLATILE RESIDUES (7 DAYS); SILICATES (28 DAYS).

3 ADD SUFFIX "D" TO BOTTLE CODE WHEN SAMPLE IS FIELD FILTERED.

4 EXCESS ACID PRESERVATIVE WILL CAUSE INTERFERENCE...COUNT DROPS CAREFULLY, CHECK pH, ADD MORE ACID IF NECESSARY (pH=2). GA INCLUDES CHEMICAL OXYGEN DEMAND (COD), TOTAL ORGANIC CARBON (TOC), NITRATE PLUS NITRITE, AMMONIA, KJELDAHL NITROGEN, AND TOTAL PHOSPHORUS.

5 CHLORINATED SAMPLES FOR PHENOLS SHOULD BE COLLECTED IN A SEPARATE BOTTLE AND DECHLORINATED WITH .141N FAS (FERROUS AMMONIUM SULFATE, USUALLY ONE DROP) BEFORE PRESERVATION (USE BOTTLE CODE GP).

6 GB INCLUDES TOTAL CYANIDE, AND CYANIDE AMMENABLE TO CHLORINATION (FREE). HOLDING TIME IS 24 HOURS IF SULFIDES ARE PRESENT.

7 CHLORINATED SAMPLES FOR CYANIDES MUST BE DECHLORINATED WITH ASCORBIC ACID (0.6 G/L) IMMEDIATELY AFTER COLLECTION AND THEN PRESERVED WITH NaOH.

8 THE PROPER CONTAINER DEPENDS ON PARAMETERS REQUESTED. 250 ML F0R TOTAL ONLY, 500 ML FOR AMENABLE

9 IF SAMPLE IS FILTERED ON SITE AND THE MEMBRANE FILTER ADDED TO 90% ACETONE (SUPPLIED BY THE LAB) AND REFRIGERATED, HOLDING TIME IS ONE MONTH. STORE FILTERED SAMPLE IN THE DARK AND ON ICE.

10 RECOMMENDED MAXIMUM HOLDING TIME FOR MERCURY, SODIUM, POTASSIUM, MAGNESIUM, CALCIUM (28 DAYS); OTHER METALS (6 MONTHS).

11 IF FIELD FILTRATION IS NOT AVAILABLE, SEND UNFILTERED SAMPLE TO THE LAB AS SOON AS POSSIBLE (WITHIN 24 HOURS). DO NOT ADD ACID TO DISSOLVED METAL IF UNFILTERED.

12 MN, MINERALS/NEUTRAL INCLUDES THE FOLLOWING PARAMETERS WITH THEIR HOLDING TIMES: pH (ANALYSES SHOULD BE PERFORMED IMMEDIATELY ON SITE); HEXAVALENT CHROMIUM (24 HOURS); ALKALINITIES (CO3, HCO3, TOTAL ALK) (14 DAYS); SPECIFIC CONDUCTANCE, CHLORIDE, SULFATE (28 DAYS).

13 FILL BOTTLE COMPLETELY (NO AIR BUBBLES) AND MAKE SURE TEFLON SIDE OF SEPTUM FACES SAMPLES. DUPLICATE VIALS REQUIRED.

14 IF BOTH ON AND BNA ARE COLLECTED,COLLECT A MAXIMUM OF 3 AMBERS PER SAMPLE IDENTIFICATION.

15 INCLUDES PETROLEUM HYDROCARBONS, SPILL IDENTIFICATION.

16 WEIGH OUT 10 GRAMS OF SOIL AND ADD TO A 40mL TARED VIAL. OPEN THE 10 mL METHANOL TUBE AND ADD TO THE VIAL. FILL, CAP, AND LABEL THE SYRINGE WITH SOIL SAMPLE.

17 LOW LEVEL PCB BY SPECIAL REQUEST ONLY. COLLECT 3 AMBERS PER SAMPLE.

18 COLLECT 3 AMBERS PER SAMPLE PER PARAMETER GROUP FOR QA/QC FOR ONE SAMPLE IN TWENTY.

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

LABORATORY SERVICES SECTION
ENVIRONMENTAL LABORATORY
QUALITY ASSURANCE PROGRAM PLAN

APPENDIX D

Annual Accuracy and Precision Statements

QAPP-Ver2.0, Revison 0
May 5, 2003

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
FULL LCS-Water

			ACCURACY				Control Windows			
			Standard	Average	Recovery		% Recovery			
Compound	TV (ug/L)	N	Deviation (ug/L)	ug/L	%	% RSD	LCL	UCL	LWL	UWL
Dichlorodifluoromethane	50	22	7.859	52.07	104.14	15.09	56.98	151.29	72.70	135.57
Chloromethane	50	22	5.809	51.84	103.68	11.21	68.83	138.54	80.44	126.92
Vinyl Chloride	50	22	6.962	54.68	109.36	12.73	67.59	151.13	81.52	137.21
Bromomethane	50	22	8.089	55.43	110.86	14.59	62.33	159.40	78.51	143.22
Chloroethane	50	22	6.201	54.25	108.50	11.43	71.30	145.70	83.70	133.30
Trichlorofluoromethane	50	22	5.718	56.00	112.00	10.21	77.69	146.31	89.13	134.87
2-Propanone (acetone)	50	22	14.693	48.41	96.82	30.35	8.66	184.98	38.04	155.59
Diethylether	50	22	5.032	55.70	111.41	9.03	81.21	141.60	91.28	131.54
1,1-Dichloroethylene	50	22	5.473	56.55	113.09	9.68	80.25	145.93	91.20	134.98
Methyl Iodide	50	22	14.774	62.52	125.05	23.63	36.40	213.69	65.95	184.14
Acrylonitrile	50	22	6.130	56.43	112.86	10.86	76.08	149.64	88.34	137.38
Methylene Chloride	50	22	5.292	54.84	109.68	9.65	77.93	141.44	88.51	130.85
Carbon Disulfide	50	22	4.968	55.02	110.05	9.03	80.24	139.85	90.17	129.92
trans-1,2-Dichloroethylene	50	22	5.060	55.43	110.86	9.13	80.50	141.22	90.62	131.10
Methyl Tertiary Butyl Ether	50	22	4.763	55.11	110.23	8.64	81.65	138.81	91.17	129.28
1,1-Dichloroethane	50	22	5.228	54.89	109.77	9.53	78.40	141.14	88.86	130.68
2-Butanone (MEK)	50	22	10.093	48.14	96.27	20.97	35.72	156.83	55.90	136.64
cis-1,2-Dichloroethylene	50	22	5.131	55.20	110.41	9.29	79.62	141.19	89.89	130.93
Chloroform	50	22	5.155	55.11	110.23	9.35	79.30	141.15	89.61	130.85
Tetrahydrofuran	50	22	5.583	54.50	109.00	10.24	75.50	142.50	86.67	131.33
Bromochloromethane	50	22	5.817	54.61	109.23	10.65	74.33	144.13	85.96	132.49
1,1,1-Trichloroethane	50	22	4.907	55.84	111.68	8.79	82.24	141.13	92.05	131.31
1,2-Dichloroethane	50	22	4.922	55.80	111.59	8.82	82.06	141.13	91.90	131.28
Benzene	50	22	4.748	54.95	109.91	8.64	81.42	138.40	90.92	128.90
Carbon Tetrachloride	50	22	5.500	56.48	112.95	9.74	79.95	145.95	90.95	134.95
1,2-Dichloropropane	50	22	4.825	55.70	111.41	8.66	82.46	140.36	92.11	130.71
Trichloroethylene	50	22	4.535	55.20	110.41	8.21	83.20	137.62	92.27	128.55
Dibromomethane	50	22	4.522	56.20	112.41	8.04	85.28	139.54	94.32	130.50
Bromodichloromethane	50	22	4.993	56.05	112.09	8.91	82.14	142.05	92.12	132.06
4-Methyl-2-Pentanone (MIBK)	50	22	5.528	54.57	109.14	10.13	75.97	142.30	87.03	131.25
cis-1,3-Dichloropropene	50	22	4.461	54.73	109.45	8.15	82.69	136.22	91.61	127.30
trans-1,3-Dichloropropene	50	22	4.816	54.93	109.86	8.77	80.97	138.76	90.60	129.13
Toluene	50	22	4.428	54.34	108.68	8.15	82.12	135.25	90.97	126.39
1,1,2-Trichloroethane	50	22	4.486	56.07	112.14	8.00	85.22	139.05	94.19	130.08
2-Hexanone	50	22	9.076	49.70	99.41	18.26	44.95	153.86	63.11	135.71
Dibromochloromethane	50	22	4.946	55.59	111.18	8.90	81.50	140.86	91.40	130.97
1,2-Dibromoethane	50	22	4.428	56.20	112.41	7.88	85.84	138.98	94.70	130.12
Tetrachloroethylene	50	22	4.457	54.48	108.95	8.18	82.21	135.70	91.12	126.78
Chlorobenzene	50	22	4.029	54.61	109.23	7.38	85.05	133.40	93.11	125.35

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
FULL LCS-Water

1,1,1,2-Tetrachloroethane	50	22	4.988	56.50	113.00	8.83	83.07	142.93	93.05	132.95

			ACCURACY				Control Windows			
				Average	Recovery		% Recovery			
Compound	TV (ug/L)	N	Standard Deviation (ug/L)	ug/L	%	% RSD	LCL	UCL	LWL	UWL
Ethylbenzene	50	22	4.464	55.00	110.00	8.12	83.22	136.78	92.14	127.86
m & P-Xylene	50	22	4.072	52.93	105.86	7.69	81.43	130.29	89.58	122.15
Bromoform	50	22	5.454	55.02	110.05	9.91	77.32	142.77	88.23	131.86
Styrene	50	22	4.731	56.61	113.23	8.36	84.84	141.61	94.30	132.15
o-Xylene	50	22	4.422	55.07	110.14	8.03	83.60	136.67	92.45	127.82
1,1,2,2-Tetrachloroethane	50	22	5.651	53.86	107.73	10.49	73.82	141.63	85.12	130.33
1,2,3-Trichloropropane	50	22	6.411	55.57	111.14	11.54	72.67	149.60	85.49	136.78
1,4-Dichloro-2-butene	50	22	8.735	46.93	93.86	18.61	41.46	146.27	58.93	128.80
Isopropylbenzene	50	22	5.674	54.57	109.14	10.40	75.09	143.18	86.44	131.83
Bromobenzene	50	22	5.309	53.39	106.77	9.95	74.92	138.63	85.54	128.01
n-Propyl Benzene	50	22	5.946	54.89	109.77	10.83	74.10	145.45	85.99	133.56
1,3,5-Trimethylbenzene	50	22	5.359	54.43	108.86	9.85	76.71	141.02	87.43	130.30
tert-Butylbenzene	50	22	5.714	54.86	109.73	10.41	75.44	144.01	86.87	132.58
1,2,4-Trimethylbenzene	50	22	4.921	53.61	107.23	9.18	77.70	136.75	87.54	126.91
sec-Butylbenzene	50	22	5.813	54.86	109.73	10.60	74.85	144.60	86.48	132.98
1,3-Dichlorobenzene	50	22	5.269	53.64	107.27	9.82	75.66	138.89	86.20	128.35
1,4-Dichlorobenzene	50	22	4.879	52.70	105.41	9.26	76.14	134.68	85.89	124.92
p-isopropyl Toluene	50	22	5.445	54.57	109.14	9.98	76.47	141.81	87.36	130.92
1,2-Dichlorobenzene	50	22	5.178	53.61	107.23	9.66	76.16	138.29	86.52	127.94
n-Butylbenzene	50	22	5.824	55.02	110.05	10.58	75.10	144.99	86.75	133.34
Hexachloroethane	50	22	6.649	53.55	107.09	12.42	67.20	146.99	80.49	133.69
1,2-Dibromo-3-chloropropane	50	22	5.816	54.02	108.05	10.77	73.15	142.94	84.78	131.31
1,2,4-Trimethylbenzene	50	22	5.507	53.39	106.77	10.32	73.73	139.82	84.74	128.80
Naphthalene	50	22	7.566	53.43	106.86	14.16	61.47	152.26	76.60	137.13
1,2,3-Trichlorobenzene	50	22	6.237	53.41	106.82	11.68	69.40	144.24	81.87	131.76
2-Methyl Naphthalene	50	22	11.241	54.73	109.45	20.54	42.01	176.90	64.49	154.42

LCS= Lab Control Sample

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
MS/MSD - Water

ACCURACY

Compound	TV (ug/L)	N	Standard Deviation (ug/L)	Average Recovery ug/L	Average Recovery %	% RSD	Control Windows % Recovery LCL	UCL	LWL	UWL
1,1-Dichloroethylene	50	765	8.099	50.08	100.16	16.17	51.57	148.75	67.76	132.55
Benzene	50	761	6.757	50.73	101.46	13.32	60.92	142.00	74.44	128.49
Trichloroethylene	50	765	6.684	48.96	97.92	13.65	57.82	138.03	71.19	124.66
Toluene	50	759	6.439	50.96	101.93	12.63	63.29	140.56	76.17	127.68
Chlorobenzene	50	765	6.102	50.16	100.32	12.17	63.71	136.94	75.91	124.73

PRECISION*

Compound		PAIRS	SD of RPDs	Average RPD	RPD	UCL		UWL
1,1-Dichloroethylene		379	9.582	7.82		28.75		19.16
Benzene		377	10.215	7.58		30.65		20.43
Trichloroethylene		379	9.930	7.79		29.79		19.86
Toluene		376	9.490	7.32		28.47		18.98
Chlorobenzene		379	9.536	7.28		28.61		19.07

***Precision is from in batch MS/MSD spike duplicates**

MS= Matrix Spike

MSD= Matrix Spike Duplicate

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

SD= Standard Deviation

RPD= Relative Percent Difference

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
FULL LIST MS/MSD - Water

			ACCURACY				Control Windows			
							% Recovery			
Compound	TV (ug/L)	N	Standard Deviation (ug/L)	Average ug/L	Recovery %	% RSD	LCL	UCL	LWL	UWL
Dichlorodifluoromethane	50	8	5.309	53.79	107.58	9.87	75.73	139.43	86.34	128.81
Chloromethane	50	8	7.608	49.52	99.03	15.36	53.38	144.68	68.60	129.46
Vinyl Chloride	50	8	6.436	51.57	103.13	12.48	64.51	141.75	77.39	128.87
Bromomethane	50	8	11.744	45.30	90.60	25.92	20.14	161.06	43.63	137.58
Chloroethane	50	8	5.447	49.93	99.85	10.91	67.17	132.53	78.06	121.64
Trichlorofluoromethane	50	8	5.239	50.46	100.91	10.38	69.48	132.35	79.96	121.87
2-Propanone (acetone)	50	8	5.853	28.35	56.69	20.65	21.57	91.81	33.28	80.10
Diethylether	50	8	6.344	50.16	100.32	12.65	62.25	138.38	74.94	125.69
1,1-Dichloroethylene	50	8	5.788	50.70	101.41	11.42	66.68	136.14	78.26	124.56
Methyl Iodide	50	8	9.032	61.50	123.01	14.68	68.82	177.20	86.88	159.14
Acrylonitrile	50	8	7.254	51.05	102.09	14.21	58.57	145.62	73.08	131.11
Methylene Chloride	50	8	7.106	48.93	97.87	14.52	55.23	140.50	69.45	126.29
Carbon Disulfide	50	8	5.607	49.84	99.68	11.25	66.03	133.32	77.25	122.11
trans-1,2-Dichloroethylene	50	8	5.327	48.55	97.10	10.97	65.14	129.06	75.79	118.41
Methyl Tertiary Butyl Ether	50	8	5.935	49.48	98.95	12.00	63.34	134.56	75.21	122.69
1,1-Dichloroethane	50	8	5.437	47.55	95.10	11.43	62.48	127.72	73.35	116.85
2-Butanone (MEK)	50	8	4.415	37.40	74.80	11.81	48.31	101.29	57.14	92.46
cis-1,2-Dichloroethylene	50	8	5.223	47.08	94.16	11.09	62.83	125.50	73.27	115.06
Chloroform	50	8	4.822	47.69	95.37	10.11	66.44	124.31	76.08	114.66
Tetrahydrofuran	50	8	7.714	48.01	96.01	16.07	49.72	142.30	65.15	126.87
Bromochloromethane	50	8	4.951	48.47	96.94	10.21	67.24	126.65	77.14	116.75
1,1,1-Trichloroethane	50	8	4.486	48.15	96.29	9.32	69.37	123.21	78.34	114.24
1,2-Dichloroethane	50	8	4.671	49.00	97.99	9.53	69.97	126.02	79.31	116.68
Benzene	50	8	4.689	48.36	96.71	9.70	68.58	124.85	77.96	115.47
Carbon Tetrachloride	50	8	5.192	47.95	95.91	10.83	64.75	127.06	75.14	116.67
1,2-Dichloropropane	50	8	4.697	48.57	97.15	9.67	68.97	125.32	78.36	115.93
Trichloroethylene	50	8	4.753	46.22	92.43	10.28	63.91	120.95	73.42	111.44
Dibromomethane	50	8	4.898	49.23	98.45	9.95	69.06	127.84	78.86	118.04
Bromodichloromethane	50	8	4.702	48.88	97.75	9.62	69.54	125.97	78.95	116.56
4-Methyl-2-Pentanone (MIBK)	50	8	6.536	49.67	99.33	13.16	60.12	138.54	73.19	125.47

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
FULL LIST MS/MSD - Water

			ACCURACY				Control Windows			
							% Recovery			
Compound	TV (ug/L)	N	Standard Deviation (ug/L)	Average ug/L	Recovery %	% RSD	LCL	UCL	LWL	UWL
cis-1,3-Dichloropropene	50	8	4.928	48.05	96.10	10.26	66.53	125.67	76.39	115.81
trans-1,3-Dichloropropene	50	8	4.884	48.47	96.94	10.08	67.64	126.24	77.40	116.47
Toluene	50	8	4.815	47.16	94.31	10.21	65.43	123.20	75.05	113.57
1,1,2-Trichloroethane	50	8	5.105	48.65	97.30	10.49	66.67	127.93	76.88	117.72
2-Hexanone	50	8	4.489	39.50	78.99	11.36	52.06	105.92	61.04	96.95
Dibromochloromethane	50	8	5.130	49.29	98.59	10.41	67.81	129.37	78.07	119.11
1,2-Dibromoethane	50	8	5.436	49.10	98.20	11.07	65.58	130.81	76.45	119.94
Tetrachloroethylene	50	8	5.359	45.12	90.24	11.88	58.08	122.39	68.80	111.67
Chlorobenzene	50	8	5.664	46.08	92.16	12.29	58.18	126.15	69.50	114.82
1,1,1,2-Tetrachloroethane	50	8	4.769	49.17	98.34	9.70	69.72	126.95	79.26	117.41
Ethylbenzene	50	8	5.645	46.74	93.49	12.08	59.62	127.36	70.91	116.07
m & P-Xylene	50	8	4.834	44.65	89.30	10.83	60.30	118.31	69.97	108.64
Bromoform	50	8	7.080	47.21	94.42	15.00	51.94	136.91	66.10	122.75
Styrene	50	8	10.036	44.63	89.26	22.49	29.04	149.47	49.11	129.40
o-Xylene	50	8	5.589	46.86	93.72	11.93	60.18	127.25	71.36	116.07
1,1,2,2-Tetrachloroethane	50	8	5.833	48.98	97.96	11.91	62.96	132.96	74.62	121.29
1,2,3-Trichloropropane	50	8	6.771	50.33	100.66	13.45	60.03	141.29	73.58	127.74
1,4-Dichloro-2-butene	50	8	7.626	45.18	90.36	16.88	44.60	136.11	59.85	120.86
Isopropylbenzene	50	8	7.406	46.40	92.80	15.96	48.37	137.24	63.18	122.43
Bromobenzene	50	8	7.067	43.91	87.83	16.09	45.43	130.23	59.56	116.10
n-Propyl Benzene	50	8	8.381	45.35	90.69	18.48	40.41	140.98	57.17	124.22
1,3,5-Trimethylbenzene	50	8	7.842	44.88	89.76	17.47	42.71	136.81	58.40	121.13
tert-Butylbenzene	50	8	8.795	45.14	90.28	19.48	37.51	143.05	55.10	125.46
1,2,4-Trimethylbenzene	50	8	7.320	44.17	88.35	16.57	44.43	132.27	59.07	117.63
sec-Butylbenzene	50	8	10.069	43.36	86.72	23.22	26.31	147.13	46.45	127.00
1,3-Dichlorobenzene	50	8	9.290	43.38	86.75	21.42	31.01	142.49	49.59	123.91
1,4-Dichlorobenzene	50	8	8.829	42.61	85.21	20.72	32.24	138.18	49.90	120.53
p-isopropyl Toluene	50	8	10.072	43.11	86.22	23.36	25.78	146.65	45.93	126.50
1,2-Dichlorobenzene	50	8	9.561	43.00	86.00	22.24	28.63	143.36	47.75	124.24
n-Butylbenzene	50	8	11.442	41.81	83.63	27.37	14.97	152.28	37.86	129.40
Hexachloroethane	50	8	12.504	40.04	80.07	31.23	5.04	155.10	30.05	130.09
1,2-Dibromo-3-chloropropane	50	8	7.793	48.99	97.97	15.91	51.22	144.73	66.80	129.14
1,2,4-Trimethylbenzene	50	8	12.946	40.09	80.18	32.29	2.51	157.86	28.40	131.97
Naphthalene	50	8	12.330	44.20	88.40	27.90	14.42	162.38	39.08	137.72
1,2,3-Trichlorobenzene	50	8	12.793	39.72	79.44	32.21	2.68	156.20	28.26	130.61
2-Methyl Naphthalene	50	8	19.401	46.01	92.03	42.16	-24.38	208.43	14.42	169.63

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
FULL LIST MS/MSD - Water

		PRECISION*			RPD		
Compound		PAIRS	SD of RPDs	Average RPD		UCL	UWL
Dichlorodifluoromethane		4	7.787	8.04		23.36	15.57
Chloromethane		4	9.526	9.26		28.58	19.05
Vinyl Chloride		4	7.893	8.73		23.68	15.79
Bromomethane		4	14.895	17.68		44.68	29.79
Chloroethane		4	8.836	10.06		26.51	17.67
Trichlorofluoromethane		4	9.962	8.55		29.89	19.92
2-Propanone (acetone)		4	15.884	21.81		47.65	31.77
Diethylether		4	10.485	9.14		31.46	20.97
1,1-Dichloroethylene		4	10.219	9.02		30.66	20.44
Methyl Iodide		4	12.622	21.56		37.86	25.24
Acrylonitrile		4	11.757	10.19		35.27	23.51
Methylene Chloride		4	9.566	8.81		28.70	19.13
Carbon Disulfide		4	9.750	8.38		29.25	19.50
trans-1,2-Dichloroethylene		4	10.163	9.10		30.49	20.33
Methyl Tertiary Butyl Ether		4	10.019	10.80		30.06	20.04
1,1-Dichloroethane		4	9.622	9.04		28.87	19.24
2-Butanone (MEK)		4	11.506	11.36		34.52	23.01
cis-1,2-Dichloroethylene		4	10.187	9.30		30.56	20.37
Chloroform		4	9.669	9.04		29.01	19.34
Tetrahydrofuran		4	10.373	11.35		31.12	20.75
Bromochloromethane		4	10.302	9.61		30.91	20.60
1,1,1-Trichloroethane		4	10.484	9.42		31.45	20.97
1,2-Dichloroethane		4	10.401	8.98		31.20	20.80
Benzene		4	10.373	9.01		31.12	20.75
Carbon Tetrachloride		4	11.411	10.36		34.23	22.82
1,2-Dichloropropane		4	10.044	8.82		30.13	20.09
Trichloroethylene		4	11.073	9.41		33.22	22.15
Dibromomethane		4	9.912	9.71		29.74	19.82
Bromodichloromethane		4	10.386	9.34		31.16	20.77
4-Methyl-2-Pentanone (MIBK)		4	12.294	11.04		36.88	24.59
cis-1,3-Dichloropropene		4	10.759	9.78		32.28	21.52
trans-1,3-Dichloropropene		4	10.461	10.23		31.38	20.92
Toluene		4	11.380	10.03		34.14	22.76
1,1,2-Trichloroethane		4	10.080	9.34		30.24	20.16
2-Hexanone		4	12.465	11.10		37.40	24.93
Dibromochloromethane		4	9.298	10.98		27.89	18.60
1,2-Dibromoethane		4	10.271	10.43		30.81	20.54
Tetrachloroethylene		4	9.753	8.82		29.26	19.51
Chlorobenzene		4	9.285	9.52		27.86	18.57
1,1,1,2-Tetrachloroethane		4	9.617	9.82		28.85	19.23
Ethylbenzene		4	10.128	9.53		30.38	20.26
m & P-Xylene		4	10.118	9.39		30.35	20.24
Bromoform		4	9.097	12.64		27.29	18.19

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
FULL LIST MS/MSD - Water

		PRECISION*			RPD		
Compound		PAIRS	SD of RPDs	Average RPD		UCL	UWL
Styrene		4	8.762	11.40		26.29	17.52
o-Xylene		4	9.829	9.73		29.49	19.66
1,1,2,2-Tetrachloroethane		4	9.628	10.29		28.88	19.26
1,2,3-Trichloropropane		4	10.558	9.75		31.67	21.12
1,4-Dichloro-2-butene		4	12.220	11.96		36.66	24.44
Isopropylbenzene		4	10.335	9.73		31.01	20.67
Bromobenzene		4	9.588	10.77		28.76	19.18
n-Propyl Benzene		4	9.424	10.56		28.27	18.85
1,3,5-Trimethylbenzene		4	9.339	10.68		28.02	18.68
tert-Butylbenzene		4	10.023	10.85		30.07	20.05
1,2,4-Trimethylbenzene		4	10.267	11.03		30.80	20.53
sec-Butylbenzene		4	9.394	11.74		28.18	18.79
1,3-Dichlorobenzene		4	10.414	11.45		31.24	20.83
1,4-Dichlorobenzene		4	9.489	10.96		28.47	18.98
p-isopropyl Toluene		4	9.843	11.27		29.53	19.69
1,2-Dichlorobenzene		4	9.678	12.14		29.03	19.36
n-Butylbenzene		4	10.053	11.95		30.16	20.11
Hexachloroethane		4	10.547	14.11		31.64	21.09
1,2-Dibromo-3-chloropropane		4	12.279	12.88		36.84	24.56
1,2,4-Trimethylbenzene		4	9.808	14.66		29.42	19.62
Naphthalene		4	10.301	16.13		30.90	20.60
1,2,3-Trichlorobenzene		4	10.006	15.80		30.02	20.01
2-Methyl Naphthalene		4	10.296	23.36		30.89	20.59

*Precision is from in batch MS/MSD spike duplicates

MS= Matrix Spike

MSD= Matrix Spike Duplicate

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

SD= Standard Deviation

RPD= Relative Percent Difference

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
LCS-Water

			ACCURACY				Control Windows			
				Average Recovery			% Recovery			
Compound	TV (ug/L)	N	Standard Deviation (ug/L)	ug/L	%	% RSD	LCL	UCL	LWL	UWL
1,1-Dichloroethylene	50	287	6.437	50.10	100.20	12.85	61.57	138.82	74.45	125.95
Benzene	50	287	5.221	50.47	100.93	10.35	69.60	132.26	80.05	121.82
Trichloroethylene	50	287	5.422	49.96	99.92	10.85	67.38	132.45	78.23	121.61
Toluene	50	287	5.126	50.70	101.39	10.11	70.64	132.14	80.89	121.89
Chlorobenzene	50	287	5.061	50.33	100.66	10.06	70.29	131.03	80.42	120.91

LCS= Lab Control Sample

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260/624
Surrogate - Water

			ACCURACY				Control Windows			
				Average	Recovery		% Recovery			
Compound	TV (ug/L)	N	Standard Deviation (ug/L)	ug/L	%	% RSD	LCL	UCL	LWL	UWL
Dibromofluoromethane	50	10850	2.523	49.75	99.50	5.07	84.36	114.65	89.41	109.60
Toluene-d8	50	10850	1.235	50.37	100.73	2.45	93.33	108.14	95.80	105.67
4-Bromofluorobenzene	50	10850	3.603	51.98	103.95	6.93	82.33	125.57	89.54	118.37

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary
Method 625
LCS - Water 2001

			ACCURACY				Control Windows			
							% Recovery			
Compound	TV (ug/L)	N	Standard Deviation (ug/L)	Average ug/L	Recovery %	% RSD	LCL	UCL	LWL	UWL
N-Nitrosodimethylamine	50	18	4.140	21.61	43.21	19.16	18.37	68.05	26.65	59.77
Phenol	100	17	5.002	27.00	27.00	18.53	11.99	42.00	16.99	37.00
Bis (2-chloroethyl) ether	50	18	6.140	29.56	59.12	20.77	22.28	95.97	34.56	83.68
2-Chlorophenol	100	17	11.925	58.94	58.94	20.23	23.17	94.72	35.09	82.79
1,3-Dichlorobenzene	50	18	4.456	18.31	36.61	24.34	9.88	63.35	18.79	54.44
1,4-Dichlorobenzene	50	18	4.413	18.92	37.84	23.33	11.36	64.32	20.18	55.49
1,2-Dichlorobenzene	50	18	4.863	20.38	40.76	23.86	11.58	69.94	21.31	60.21
Bis (2-chloroisopropyl) ether	50	18	6.365	30.58	61.16	20.81	22.97	99.35	35.70	86.62
Hexachloroethane	50	18	11.077	24.60	49.19	45.03	-17.27	115.65	4.89	93.50
N-nitrosodi-n-propyl amine	50	18	9.666	27.88	55.76	34.67	-2.23	113.75	17.10	94.42
Nitrobenzene	50	18	6.432	31.62	63.25	20.34	24.66	101.84	37.52	88.97
Isophorone	50	18	5.569	31.00	61.99	17.97	28.57	95.41	39.71	84.27
2 - Nitrophenol	100	17	12.878	63.69	63.69	20.22	25.06	102.33	37.93	89.45
2,4 - Dimethylphenol	100	17	11.559	65.50	65.50	17.65	30.82	100.17	42.38	88.62
Bis (2-chloroethoxy) methane	50	18	6.385	33.75	67.49	18.92	29.18	105.80	41.95	93.03
2,4 - Dichlorophenol	100	17	12.611	68.31	68.31	18.46	30.48	106.14	43.09	93.53
1,2,4 - Trichlorobenzene	50	18	4.899	21.61	43.22	22.67	13.83	72.62	23.63	62.82
Naphthalene	50	18	5.305	25.29	50.58	20.98	18.74	82.41	29.35	71.80
Hexachlorobutadiene	50	18	5.061	19.95	39.90	25.37	9.54	70.27	19.66	60.15
Phenol, 4-chloro-3-methyl-	50	18	19.883	68.42	136.85	29.06	17.55	256.15	57.31	216.38
Hexachlorocyclopentadiene	50	18	3.565	12.41	24.82	28.73	3.43	46.22	10.56	39.08
2,4,6 - Trichlorophenol	100	17	12.323	71.40	71.40	17.26	34.43	108.37	46.76	96.05
2- Chloronaphthalene	50	18	5.720	27.18	54.36	21.05	20.04	88.68	31.48	77.24
Dimethylphthalate	50	18	7.793	33.96	67.92	22.95	21.16	114.67	36.75	99.09
Acenaphthylene	50	18	7.587	33.03	66.06	22.97	20.54	111.58	35.71	96.41
2,6 - Dinitrotoluene	50	18	5.632	37.17	74.34	15.15	40.55	108.13	51.81	96.86
Acenaphthene	50	18	5.674	29.79	59.57	19.05	25.53	93.61	36.88	82.27
2,4 - Dinitrophenol	100	17	10.056	63.37	63.37	15.87	33.20	93.53	43.25	83.48
2,4 - Dinitrotoluene	50	18	5.478	34.51	69.02	15.87	36.15	101.89	47.11	90.93
4-Nitrophenol	100	18	10.487	32.04	32.04	32.73	0.58	63.50	11.07	53.01
Fluorene	50	18	6.036	31.85	63.70	18.95	27.48	99.92	39.55	87.85
Diethylphthalate	50	18	6.072	37.61	75.23	16.14	38.79	111.66	50.94	99.52
4-Chlorophenyl phenyl ether	50	18	5.828	30.45	60.89	19.14	25.93	95.86	37.58	84.21
4,6 - Dinitro-2-methylphenol	100	17	9.199	75.70	75.70	12.15	48.10	103.30	57.30	94.10
N-Nitrosodiphenylamine	50	18	6.141	38.31	76.62	16.03	39.78	113.47	52.06	101.19
4-Bromophenyl phenyl ether	50	18	5.358	35.57	71.13	15.07	38.98	103.28	49.70	92.57
Hexachlorobenzene	50	18	5.437	38.25	76.50	14.21	43.88	109.12	54.75	98.25
Pentachlorophenol	100	17	9.372	71.79	71.79	13.05	43.68	99.91	53.05	90.54
Phenanthrene	50	18	5.288	37.06	74.12	14.27	42.40	105.85	52.97	95.27
Anthracene	50	18	4.607	35.48	70.95	12.99	43.31	98.60	52.53	89.38
Di-n-butyl phthalate	50	18	6.186	40.79	81.58	15.16	44.47	118.70	56.84	106.32
Fluoranthene	50	18	5.607	40.15	80.29	13.97	46.65	113.94	57.86	102.72
Pyrene	50	18	5.583	38.87	77.73	14.37	44.23	111.23	55.40	100.07
Butylbenzyl phthalate	50	18	7.608	40.53	81.07	18.77	35.42	126.72	50.64	111.50
Benzo [a] anthracene	50	18	4.935	40.40	80.79	12.22	51.18	110.40	61.05	100.53
Chrysene	50	18	5.052	41.03	82.07	12.31	51.76	112.38	61.86	102.28
Bis (2-ethylhexyl) phthalate	50	18	58.332	56.13	112.27	103.92	-237.73	462.26	-121.06	345.59
Di-n-Octyl phthalate	50	18	4.517	40.62	81.23	11.12	54.13	108.34	63.16	99.30
Benzo [b] fluoranthene	50	18	6.396	42.74	85.48	14.97	47.10	123.86	59.89	111.06
Benzo [k] fluoranthene	50	18	5.864	41.24	82.48	14.22	47.30	117.66	59.03	105.93
Benzo [a] pyrene	50	18	4.613	36.89	73.78	12.50	46.10	101.46	55.33	92.23
Indeno (1,2,3-cd) pyrene	50	18	7.897	37.76	75.52	20.92	28.13	122.90	43.93	107.11
Dibenzo [a,h] anthracene	50	18	7.266	38.07	76.14	19.09	32.55	119.74	47.08	105.21
Benzo [g,h,i] perylene	50	18	7.056	38.64	77.28	18.26	34.95	119.62	49.06	105.51

LCS = Lab Control Sample
TV= True Value
N= Number of data points
RSD= Relative Standard Deviation

LCL= Lower Control Limit
UCL= Upper Control Limit
LWL= Lower Warning Limit
UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary
Method 8270
MS/MSD - Water
2001

ACCURACY

Compound	TV (ug/L)	N	Standard Deviation (ug/L)	Average Recovery ug/L	Average Recovery %	% RSD	Control Windows % Recovery LCL	UCL	LWL	UWL
1,4-Dichlorobenzene	50	152	7.073	22.32	44.64	31.69	2.20	87.08	16.35	72.93
N-Nitroso-Di-N-Propylamine	50	152	10.037	34.71	69.42	28.91	9.20	129.65	29.28	109.57
1,2,4-Trichlorobenzene	50	152	7.860	25.98	51.97	30.25	4.81	99.13	20.53	83.41
Acenaphthene	50	152	8.962	33.74	67.49	26.56	13.71	121.26	31.64	103.34
2,4-Dinitrotoluene	50	152	6.428	38.45	76.90	16.72	38.33	115.47	51.19	102.61
Pyrene	50	152	7.878	37.74	75.47	20.88	28.20	122.74	43.96	106.98
Phenol	100	148	13.392	38.61	38.61	34.68	-1.56	78.79	11.83	65.39
2-Chlorophenol	100	148	17.834	60.12	60.12	29.66	6.62	113.62	24.45	95.79
4-Chloro-m-cresol	100	148	16.338	74.30	74.30	21.99	25.28	123.31	41.62	106.97
4-Nitrophenol	100	148	14.199	50.71	50.71	28.00	8.12	93.31	22.32	79.11
Pentachlorophenol	100	148	14.228	80.95	80.95	17.58	38.26	123.63	52.49	109.41

PRECISION*

Compound		PAIRS	SD of RPDs	Average RPD	RPD	UCL		UWL
1,4-Dichlorobenzene		76	8.938	9.79		26.81		17.88
N-Nitroso-Di-N-Propylamine		76	8.613	9.13		25.84		17.23
1,2,4-Trichlorobenzene		76	8.477	8.72		25.43		16.95
Acenaphthene		76	6.751	7.22		20.25		13.50
2,4-Dinitrotoluene		76	5.953	6.21		17.86		11.91
Pyrene		76	8.410	6.83		25.23		16.82
Phenol		74	7.761	8.76		23.28		15.52
2-Chlorophenol		74	8.262	8.23		24.79		16.52
4-Chloro-m-cresol		74	5.077	6.26		15.23		10.15
4-Nitrophenol		74	16.604	11.79		49.81		33.21
Pentachlorophenol		74	22.572	10.22		67.72		45.14

***Precision is from in batch MS/MSD spike duplicates**

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

SD= Standard Deviation

RPD = Relative Percent Difference

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary
Method 8270
LCS/LCSD - Water
2001

			ACCURACY				Control Windows			
			Standard	Average Recovery			% Recovery			
Compound	TV (ug/L)	N	Deviation (ug/L)	ug/L	%	% RSD	LCL	UCL	LWL	UWL
1,4-Dichlorobenzene	50	109	4.778	18.16	36.33	26.30	7.66	64.99	17.21	55.44
N-Nitroso-Di-N-Propylamine	50	109	9.277	32.58	65.15	28.48	9.49	120.81	28.04	102.26
1,2,4-Trichlorobenzene	50	109	5.206	21.55	43.10	24.16	11.86	74.34	22.27	63.92
Acenaphthene	50	109	8.284	29.46	58.92	28.12	9.22	108.62	25.79	92.06
2,4-Dinitrotoluene	50	109	10.435	33.38	66.76	31.26	4.15	129.37	25.02	108.50
Pyrene	50	109	10.109	34.16	68.33	29.59	7.67	128.99	27.89	108.77
Phenol	100	101	5.521	26.75	26.75	20.64	10.19	43.31	15.71	37.79
2-Chlorophenol	100	101	11.216	59.48	59.48	18.86	25.83	93.13	37.05	81.91
4-Chloro-m-cresol	100	101	13.028	70.91	70.91	18.37	31.82	109.99	44.85	96.96
4-Nitrophenol	100	101	7.019	31.74	31.74	22.12	10.68	52.79	17.70	45.78
Pentachlorophenol	100	101	14.179	75.60	75.60	18.75	33.07	118.14	47.25	103.96

			PRECISION*			RPD		
Compound		PAIRS	SD of RPDs	Average RPD		UCL		UWL
1,4-Dichlorobenzene		15	9.863	11.53		29.59		19.73
N-Nitroso-Di-N-Propylamine		15	10.689	11.35		32.07		21.38
1,2,4-Trichlorobenzene		15	8.132	10.36		24.40		16.26
Acenaphthene		15	8.831	7.52		26.49		17.66
2,4-Dinitrotoluene		15	12.766	9.65		38.30		25.53
Pyrene		15	13.019	7.73		39.06		26.04
Phenol		12	7.322	7.93		21.96		14.64
2-Chlorophenol		12	5.446	7.60		16.34		10.89
4-Chloro-m-cresol		12	7.609	9.76		22.83		15.22
4-Nitrophenol		12	12.843	10.98		38.53		25.69
Pentachlorophenol		12	16.940	13.45		50.82		33.88

***Precision is from in batch LCS/LCSD spike duplicates.**

LCS = Lab Control Sample

LCSD = Lab Control Sample Duplicate

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary
Method 625
MS/MSD - Water
2001

			ACCURACY				Control Windows			
			Standard	Average	Recovery		% Recovery			
Compound	TV (ug/L)	N	Deviation (ug/L)	ug/L	%	% RSD	LCL	UCL	LWL	UWL
N-Nitrosodimethylamine	50	32	9.070	24.37	48.74	37.21	-5.68	103.16	12.46	85.02
Phenol	50	30	15.675	34.52	69.04	45.41	-25.01	163.09	6.34	131.74
Bis (2-chloroethyl) ether	50	32	9.642	27.25	54.50	35.39	-3.36	112.35	15.93	93.06
2-Chlorophenol	100	30	19.141	54.24	54.24	35.29	-3.19	111.66	15.95	92.52
1,3-Dichlorobenzene	50	32	7.786	19.46	38.93	40.00	-7.79	85.64	7.78	70.07
1,4-Dichlorobenzene	50	32	7.994	20.31	40.62	39.36	-7.34	88.59	8.65	72.60
1,2-Dichlorobenzene	100	32	8.297	21.55	21.55	38.51	-3.34	46.44	4.95	38.14
Bis (2-chloroisopropyl) ether	50	32	9.954	29.09	58.17	34.22	-1.55	117.90	18.36	97.99
Hexachloroethane	50	32	11.430	23.52	47.03	48.60	-21.55	115.61	1.31	92.75
N-nitrosodi-n-propyl amine	50	32	10.272	27.01	54.03	38.03	-7.60	115.65	12.94	95.11
Nitrobenzene	50	32	9.698	29.43	58.86	32.95	0.68	117.05	20.07	97.65
Isophorone	50	32	8.914	27.70	55.39	32.18	1.91	108.88	19.74	91.05
2 - Nitrophenol	100	30	18.373	57.97	57.97	31.70	2.85	113.09	21.22	94.71
2,4 - Dimethylphenol	100	30	17.611	62.57	62.57	28.15	9.74	115.40	27.35	97.79
Bis (2-chloroethoxy) methane	50	32	9.775	30.36	60.72	32.20	2.07	119.37	21.62	99.82
2,4 - Dichlorophenol	100	30	18.029	63.54	63.54	28.38	9.45	117.63	27.48	99.60
1,2,4 - Trichlorobenzene	50	32	8.377	23.68	47.36	35.37	-2.90	97.62	13.86	80.87
Naphthalene	50	32	8.459	26.12	52.24	32.39	1.48	103.00	18.40	86.08
Hexachlorobutadiene	50	32	7.788	22.01	44.01	35.39	-2.72	90.74	12.86	75.17
Phenol, 4-chloro-3-methyl-	100	30	16.047	69.33	69.33	23.14	21.19	117.47	37.24	101.43
Hexachlorocyclopentadiene	50	32	5.560	13.64	27.29	40.75	-6.07	60.65	5.05	49.53
2,4,6 - Trichlorophenol	100	30	16.327	67.28	67.28	24.27	18.30	116.25	34.62	99.93
2- Chloronaphthalene	50	32	8.534	28.52	57.05	29.92	5.84	108.25	22.91	91.18
Dimethylphthalate	50	32	8.247	32.86	65.71	25.10	16.23	115.19	32.73	98.70
Acenaphthylene	50	32	8.746	31.88	63.77	27.43	11.29	116.24	28.78	98.75
2,6 - Dinitrotoluene	50	32	6.996	35.56	71.12	19.68	29.14	113.09	43.13	99.10
Acenaphthene	50	32	7.809	30.65	61.30	25.48	14.44	108.15	30.06	92.53
2,4 - Dinitrophenol	100	30	14.875	57.46	57.46	25.89	12.84	102.09	27.71	87.21
2,4 - Dinitrotoluene	50	32	9.863	39.43	78.87	25.01	19.69	138.05	39.42	118.32
4-Nitrophenol	100	30	13.235	40.68	40.68	32.54	0.97	80.38	14.21	67.14
Fluorene	50	32	7.164	32.45	64.91	22.07	21.92	107.89	36.25	93.56
Diethylphthalate	50	32	6.020	36.14	72.28	16.66	36.16	108.40	48.20	96.36
4-Chlorophenyl phenyl ether	50	32	7.282	32.01	64.01	22.75	20.32	107.71	34.89	93.14
4,6 - Dinitro-2-methylphenol	100	30	16.133	70.66	70.66	22.83	22.26	119.06	38.39	102.92
N-Nitrosodiphenylamine	50	32	6.686	38.04	76.07	17.58	35.96	116.19	49.33	102.82
4-Bromophenyl phenyl ether	50	32	6.513	36.86	73.71	17.67	34.63	112.79	47.66	99.76
Hexachlorobenzene	50	32	9.349	39.23	78.46	23.83	22.37	134.55	41.07	115.85
Pentachlorophenol	100	30	14.067	72.41	72.41	19.43	30.21	114.61	44.27	100.54
Phenanthrene	50	32	5.506	37.25	74.50	14.78	41.46	107.53	52.47	96.52
Anthracene	50	32	5.723	35.07	70.14	16.32	35.80	104.48	47.25	93.03
Di-n-butyl phthalate	50	32	7.019	39.16	78.33	17.92	36.22	120.44	50.25	106.40
Fluoranthene	50	32	6.268	38.72	77.44	16.19	39.83	115.05	52.37	102.51
Pyrene	50	32	5.511	38.15	76.29	14.45	43.23	109.36	54.25	98.33
Butylbenzyl phthalate	50	32	7.315	40.13	80.25	18.23	36.36	124.14	50.99	109.51
Benzo [a] anthracene	50	32	6.413	38.67	77.35	16.58	38.87	115.82	51.70	103.00
Chrysene	50	32	6.988	39.87	79.75	17.52	37.82	121.67	51.80	107.70
Bis (2-ethylhexyl) phthalate	50	32	30.480	44.61	89.22	68.33	-93.66	272.10	-32.70	211.14
Di-n-Octyl phthalate	50	32	8.453	37.75	75.50	22.39	24.78	126.22	41.69	109.31
Benzo [b] fluoranthene	50	32	8.415	40.13	80.26	20.97	29.77	130.75	46.60	113.92
Benzo [k] fluoranthene	50	32	7.820	38.32	76.65	20.41	29.73	123.56	45.37	107.92
Benzo [a] pyrene	50	32	6.679	34.49	68.99	19.36	28.92	109.06	42.28	95.70
Indeno (1,2,3-cd) pyrene	50	32	11.330	33.88	67.77	33.44	-0.21	135.75	22.45	113.09
Dibenzo [a,h] anthracene	50	32	10.353	33.79	67.59	30.63	5.47	129.70	26.18	109.00
Benzo [g,h,i] perylene	50	31	11.949	33.93	67.85	35.22	-3.84	139.54	20.06	115.65

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary
Method 625
MS/MSD - Water
2001

		PRECISION*		RPD		
Compound	PAIRS	SD of RPDs	Average RPD		UCL		UWL
N-Nitrosodimethylamine	16	11.223	11.91		33.67		22.45
Phenol	15	8.524	10.56		25.57		17.05
Bis (2-chloroethyl) ether	16	11.455	11.61		34.36		22.91
2-Chlorophenol	15	11.335	10.42		34.01		22.67
1,3-Dichlorobenzene	16	9.477	10.05		28.43		18.95
1,4-Dichlorobenzene	16	10.869	12.10		32.61		21.74
1,2-Dichlorobenzene	16	6.867	8.73		20.60		13.73
Bis (2-chloroisopropyl) ether	16	11.553	12.04		34.66		23.11
Hexachloroethane	16	11.045	11.53		33.14		22.09
N-nitrosodi-n-propyl amine	16	9.919	9.32		29.76		19.84
Nitrobenzene	16	11.145	9.32		33.44		22.29
Isophorone	16	10.088	9.69		30.26		20.18
2 - Nitrophenol	15	10.528	8.76		31.59		21.06
2,4 - Dimethylphenol	15	8.729	7.83		26.19		17.46
Bis (2-chloroethoxy) methane	16	10.117	8.49		30.35		20.23
2,4 - Dichlorophenol	15	8.835	7.76		26.51		17.67
1,2,4 - Trichlorobenzene	16	9.560	10.04		28.68		19.12
Naphthalene	16	8.920	9.21		26.76		17.84
Hexachlorobutadiene	16	11.012	10.30		33.04		22.02
Phenol, 4-chloro-3-methyl-	15	5.748	6.38		17.24		11.50
Hexachlorocyclopentadiene	16	11.519	16.57		34.56		23.04
2,4,6 - Trichlorophenol	15	5.297	7.85		15.89		10.59
2- Chloronaphthalene	16	8.771	8.82		26.31		17.54
Dimethylphthalate	16	7.273	8.83		21.82		14.55
Acenaphthylene	16	7.117	7.98		21.35		14.23
2,6 - Dinitrotoluene	16	5.114	7.54		15.34		10.23
Acenaphthene	16	6.433	7.69		19.30		12.87
2,4 - Dinitrophenol	15	10.857	11.48		32.57		21.71
2,4 - Dinitrotoluene	16	7.709	8.74		23.13		15.42
4-Nitrophenol	15	13.298	13.93		39.89		26.60
Fluorene	16	6.881	7.67		20.64		13.76
Diethylphthalate	16	6.959	8.49		20.88		13.92
4-Chlorophenyl phenyl ether	16	7.392	7.62		22.18		14.78
4,6 - Dinitro-2-methylphenol	15	7.148	7.86		21.45		14.30
N-Nitrosodiphenylamine	16	7.275	7.74		21.83		14.55
4-Bromophenyl phenyl ether	16	5.913	6.63		17.74		11.83
Hexachlorobenzene	16	17.317	10.49		51.95		34.63
Pentachlorophenol	15	17.271	13.02		51.81		34.54
Phenanthrene	16	7.053	6.88		21.16		14.11
Anthracene	16	10.934	10.37		32.80		21.87
Di-n-butyl phthalate	16	10.387	10.31		31.16		20.77
Fluoranthene	16	8.141	8.11		24.42		16.28
Pyrene	16	8.867	9.83		26.60		17.73
Butylbenzyl phthalate	16	7.363	8.59		22.09		14.73
Benzo [a] anthracene	16	7.146	8.56		21.44		14.29
Chrysene	16	7.562	9.43		22.69		15.12
Bis (2-ethylhexyl) phthalate	16	32.495	18.59		97.49		64.99
Di-n-Octyl phthalate	16	5.942	9.19		17.83		11.88
Benzo [b] fluoranthene	16	6.350	8.10		19.05		12.70
Benzo [k] fluoranthene	16	5.754	6.83		17.26		11.51
Benzo [a] pyrene	16	6.626	9.41		19.88		13.25
Indeno (1,2,3-cd) pyrene	16	8.756	10.41		26.27		17.51
Dibenzo [a,h] anthracene	16	8.172	8.36		24.52		16.34
Benzo [g,h,i] perylene	16	48.263	22.22		144.79		96.53

***Precision is from in batch MS/MSD spike duplicates**
TV= True Value
N= Number of data points
RSD= Relative Standard Deviation
SD= Standard Deviation

RPD = Relative Percent Difference
LCL= Lower Control Limit
UCL= Upper Control Limit
LWL= Lower Warning Limit
UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8270
Surrogate - Water

			ACCURACY				Control Windows			
			Standard	Average Recovery			% Recovery			
Compound	TV (ug/L)	N	Deviation (ug/L)	ug/L	%	% RSD	LCL	UCL	LWL	UWL
2-Fluorophenol	50	873	5.862	16.63	33.25	35.25	-1.92	68.42	9.81	56.70
Phenol -d5	50	873	5.100	13.40	26.80	38.05	-3.80	57.41	6.40	47.20
Nitrobenzene -d5	25	1111	4.510	16.66	66.66	27.07	12.53	120.78	30.58	102.74
2-Fluorobiphenyl	25	1113	3.883	16.63	66.52	23.35	19.92	113.11	35.45	97.58
2,4,6-Tribromophenol	50	877	11.101	40.84	81.68	27.18	15.07	148.29	37.28	126.09
p-Terphenyl-d14	25	1113	4.320	21.63	86.52	19.97	34.68	138.37	51.96	121.09

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8081/8082/608
Surrogate - Water

			ACCURACY				Control Windows			
				Average	Recovery		% Recovery			
Compound	TV (ug/L)	N	Standard Deviation (ug/L)	ug/L	%	% RSD	LCL	UCL	LWL	UWL
Tetrachloro-m-Xylene	0.20	1047	0.026	0.097	48.38	26.87	9.39	87.37	22.38	74.37
Decachlorobiphenyl	0.20	1047	0.037	0.113	56.72	32.65	1.16	112.28	19.68	93.76

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8081/8082/608
LCS-Water

			ACCURACY				Control Windows			
			Standard	Average	Recovery		% Recovery			
Compound	TV (ug/L)	N	Deviation (ug/L)	ug/L	%	% RSD	LCL	UCL	LWL	UWL
a-BHC	0.20	64	0.023	0.144	72.12	16.066	37.359	106.877	48.945	95.291
b-BHC	0.20	64	0.024	0.164	82.24	14.505	46.455	118.033	58.385	106.103
g-BHC	0.20	64	0.025	0.150	75.06	16.580	37.723	112.389	50.167	99.945
d-BHC	0.20	64	0.023	0.156	77.96	14.509	44.025	111.894	55.337	100.582
Heptachlor	0.20	64	0.021	0.115	57.41	18.563	25.442	89.388	36.099	78.730
Aldrin	0.20	64	0.021	0.097	48.59	21.395	17.404	79.781	27.800	69.385
Hept-Epox.	0.20	64	0.028	0.159	79.57	17.844	36.973	122.158	51.171	107.961
g-Chlordane	0.20	64	0.022	0.144	72.11	15.380	38.836	105.375	49.926	94.285
Endosulfan I	0.20	64	0.038	0.128	63.97	29.819	6.744	121.203	25.821	102.126
a-Chlordane	0.20	63	0.025	0.144	71.82	17.292	34.562	109.078	46.981	96.658
Dieldrin	0.20	64	0.031	0.165	82.69	18.936	35.714	129.658	51.371	114.000
4,4'-DDE	0.20	64	0.019	0.141	70.38	13.800	41.240	99.513	50.952	89.801
Endrin	0.20	64	0.032	0.182	90.81	17.454	43.262	138.368	59.113	122.517
Endosulfan II	0.20	62	0.036	0.139	69.42	26.013	15.244	123.590	33.302	105.532
4,4'-DDD	0.20	64	0.025	0.170	84.85	14.464	48.030	121.667	60.303	109.394
Endrin Aldehyde	0.20	62	0.027	0.126	63.10	21.454	22.486	103.708	36.023	90.171
Endosulfan Sulfate	0.20	62	0.031	0.134	66.96	23.197	20.362	113.559	35.895	98.026
4,4'-DDT	0.20	64	0.024	0.152	75.99	15.833	39.896	112.085	51.927	100.054
Endrin Ketone	0.20	62	0.027	0.155	77.74	17.366	37.239	118.236	50.738	104.736
HBB	0.20	64	0.032	0.138	68.97	22.867	21.655	116.284	37.427	100.513
Methoxychlor	0.20	64	0.030	0.159	79.74	18.683	35.047	124.439	49.946	109.540
Mirex	0.20	64	0.026	0.159	79.52	16.339	40.545	118.504	53.538	105.510
BP-6	0.20	64	0.028	0.150	75.03	18.771	32.780	117.283	46.863	103.199
Aroclor 1016	1.00	22	0.102	0.635	63.45	16.096	32.812	94.088	43.024	83.876
Aroclor 1260	1.00	22	0.133	0.758	75.75	17.589	35.780	115.720	49.103	102.397

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8081/8082/608
MS/MSD - Water

			ACCURACY				Control Windows			
							% Recovery			
Compound	TV (ug/L)	N	Standard Deviation (ug/L)	Average ug/L	Recovery %	% RSD	LCL	UCL	LWL	UWL
a-BHC	0.20	86	0.025	0.133	66.74	18.636	29.428	104.061	41.867	91.622
b-BHC	0.20	88	0.024	0.156	78.13	15.346	42.162	114.103	54.152	102.113
g-BHC	0.20	88	0.027	0.141	70.50	19.265	29.756	111.251	43.339	97.668
d-BHC	0.20	88	0.022	0.149	74.29	14.653	41.633	106.950	52.519	96.064
Heptachlor	0.20	88	0.027	0.124	62.12	21.404	22.233	102.011	35.529	88.715
Aldrin	0.20	88	0.025	0.111	55.68	22.392	18.277	93.086	30.746	80.618
Hept-Epox.	0.20	88	0.025	0.155	77.36	15.950	40.346	114.380	52.685	102.041
g-Chlordane	0.20	88	0.024	0.152	75.88	16.107	39.212	112.539	51.433	100.318
Endosulfan I	0.20	88	0.033	0.120	59.97	27.457	10.574	109.375	27.041	92.908
a-Chlordane	0.20	88	0.026	0.145	72.71	17.973	33.506	111.916	46.574	98.848
Dieldrin	0.20	86	0.029	0.159	79.38	18.071	36.346	122.412	50.691	108.068
4,4'-DDE	0.20	88	0.022	0.152	75.86	14.719	42.361	109.354	53.526	98.188
Endrin	0.20	88	0.038	0.175	87.55	21.683	30.600	144.504	49.584	125.520
Endosulfan II	0.20	84	0.033	0.130	64.86	25.083	16.054	113.672	32.324	97.403
4,4'-DDD	0.20	86	0.026	0.164	82.15	15.690	43.480	120.812	56.368	107.924
Endrin Aldehyde	0.20	84	0.027	0.124	62.16	21.318	22.409	101.921	35.661	88.669
Endosulfan Sulfate	0.20	84	0.030	0.124	62.04	24.386	16.652	107.422	31.780	92.294
4,4'-DDT	0.20	88	0.028	0.148	73.83	18.647	32.527	115.125	46.293	101.359
Endrin Ketone	0.20	84	0.029	0.150	74.89	19.681	30.671	119.100	45.409	104.362
HBB	0.20	88	0.035	0.134	66.76	26.081	14.525	118.997	31.937	101.585
Methoxychlor	0.20	86	0.029	0.150	75.02	19.450	31.244	118.789	45.835	104.198
Mirex	0.20	88	0.031	0.163	81.30	19.134	34.634	127.971	50.190	112.415
BP-6	0.20	88	0.033	0.145	72.57	22.988	22.521	122.615	39.204	105.933
Aroclor 1016	1.00	10	0.111	0.734	73.36	15.135	40.050	106.670	51.153	95.567
Aroclor 1260	1.00	12	0.124	0.860	86.01	14.469	48.674	123.343	61.119	110.898

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8081/8082/608
MS/MSD - Water

			PRECISION*			RPD		
Compound		PAIRS	SD of RPDs	Average RPD		UCL		UWL
a-BHC		43	7.622	9.343		22.866		15.244
b-BHC		44	4.770	6.420		14.309		9.539
g-BHC		44	7.982	9.056		23.945		15.963
d-BHC		44	5.747	7.124		17.242		11.495
Heptachlor		44	8.537	10.427		25.612		17.074
Aldrin		44	9.415	11.306		28.246		18.831
Hept-Epox.		44	5.282	6.703		15.845		10.563
g-Chlordane		44	7.377	7.856		22.130		14.753
Endosulfan I		44	7.054	7.753		21.162		14.108
a-Chlordane		44	11.398	8.664		34.194		22.796
Dieldrin		43	5.742	6.541		17.225		11.483
4,4'-DDE		44	7.788	7.762		23.365		15.577
Endrin		44	26.403	11.750		79.209		52.806
Endosulfan II		42	5.234	6.271		15.703		10.469
4,4'-DDD		43	5.262	6.173		15.787		10.525
Endrin Aldehyde		42	7.617	8.367		22.851		15.234
Endosulfan Sulfate		42	7.016	7.026		21.048		14.032
4,4'-DDT		44	4.983	6.690		14.950		9.967
Endrin Ketone		42	5.976	6.143		17.927		11.951
HBB		44	7.498	8.104		22.495		14.997
Methoxychlor		43	9.900	8.978		29.699		19.799
Mirex		44	10.059	8.545		30.177		20.118
BP-6		44	10.043	9.141		30.128		20.085
Aroclor 1016		5	3.382	7.816		10.145		6.763
Aroclor 1260		6	4.579	7.474		13.738		9.158

***Precision is from in batch MS/MSD spike duplicates**

MS= Matrix Spike

MSD= Matrix Spike Duplicate

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

SD= Standard Deviation

RPD= Relative Percent Difference

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8081/8082
Surrogate - Soil

			ACCURACY				Control Windows			
				Average Recovery			% Recovery			
Compound	TV (ug/Kg)	N	Standard Deviation (ug/Kg)	ug/Kg	%	% RSD	LCL	UCL	LWL	UWL
Tetrachloro-m-Xylene	100	1398	15.636	76.78	76.78	20.36	29.87	123.69	45.51	108.06
Decachlorobiphenyl	100	1426	19.189	78.57	78.57	24.42	21.00	136.14	40.19	116.95

TV= True Value
N= Number of data points
RSD= Relative Standard Deviation

LCL= Lower Control Limit
UCL= Upper Control Limit
LWL= Lower Warning Limit
UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8081/8082
LCS-Soil

			ACCURACY				Control Windows			
			Standard	Average	Recovery		% Recovery			
Compound	TV (ug/Kg)	N	Deviation (ug/Kg)	ug/Kg	%	% RSD	LCL	UCL	LWL	UWL
a-BHC	100	46	12.383	78.96	78.96	15.68	41.81	116.10	54.19	103.72
b-BHC	100	46	13.873	94.11	94.11	14.74	52.49	135.73	66.36	121.85
g-BHC	100	46	14.344	83.63	83.63	17.15	40.60	126.66	54.94	112.32
d-BHC	100	45	27.333	61.33	61.33	44.56	-20.67	143.33	6.67	116.00
Heptachlor	100	46	16.386	92.41	92.41	17.73	43.25	141.57	59.64	125.19
Aldrin	100	46	12.122	85.63	85.63	14.16	49.26	122.00	61.39	109.87
Hept-Epox.	100	46	13.310	96.67	96.67	13.77	56.74	136.60	70.05	123.29
g-Chlordane	100	46	12.379	91.52	91.52	13.53	54.39	128.66	66.76	116.28
Endosulfan I	100	45	21.656	31.90	31.90	67.89	-33.07	96.87	-11.41	75.21
a-Chlordane	100	46	12.276	90.57	90.57	13.55	53.74	127.39	66.01	115.12
Dieldrin	100	46	13.423	93.50	93.50	14.36	53.23	133.77	66.65	120.35
4,4'-DDE	100	46	12.855	94.78	94.78	13.56	56.22	133.35	69.07	120.49
Endrin	100	45	14.820	106.91	106.91	13.86	62.45	151.37	77.27	136.55
Endosulfan II	100	45	22.750	37.91	37.91	60.01	-30.34	106.16	-7.59	83.41
4,4'-DDD	100	35	14.980	99.66	99.66	15.03	54.72	144.60	69.70	129.62
Endrin Aldehyde	100	35	19.002	56.57	56.57	33.59	-0.43	113.58	18.57	94.58
Endosulfan Sulfate	100	46	13.545	94.48	94.48	14.34	53.84	135.11	67.39	121.57
4,4'-DDT	100	45	23.913	102.18	102.18	23.40	30.44	173.92	54.35	150.00
Endrin Ketone	100	46	17.505	94.11	94.11	18.60	41.59	146.62	59.10	129.12
HBB	100	46	22.444	78.39	78.39	28.63	11.06	145.72	33.50	123.28
Methoxychlor	100	42	20.244	101.62	101.62	19.92	40.89	162.35	61.13	142.11
Mirex	100	46	12.547	93.76	93.76	13.38	56.12	131.40	68.67	118.86
BP-6	100	46	19.642	100.37	100.37	19.57	41.44	159.29	61.09	139.65
Aroclor 1016	500	29	71.479	465.14	93.03	15.37	50.14	135.92	64.44	121.62
Aroclor 1260	500	29	70.227	516.10	103.22	13.61	61.08	145.36	75.13	131.31

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8081/8082
MS/MSD - Soil

			ACCURACY				Control Windows			
							% Recovery			
Compound	TV (ug/Kg)	N	Standard Deviation (ug/Kg)	Average ug/Kg	Recovery %	% RSD	LCL	UCL	LWL	UWL
a-BHC	100	90	19.375	80.87	80.87	23.96	22.74	138.99	42.12	119.62
b-BHC	100	90	14.974	90.37	90.37	16.57	45.44	135.29	60.42	120.32
g-BHC	100	90	17.390	82.07	82.07	21.19	29.90	134.24	47.29	116.85
d-BHC	100	88	26.399	71.30	71.30	37.03	-7.90	150.49	18.50	124.09
Heptachlor	100	87	15.454	89.34	89.34	17.30	42.98	135.71	58.44	120.25
Aldrin	100	90	14.081	83.06	83.06	16.95	40.81	125.30	54.89	111.22
Hept-Epox.	100	90	13.088	89.99	89.99	14.54	50.73	129.25	63.81	116.16
g-Chlordane	100	90	13.956	83.69	83.69	16.68	41.82	125.56	55.78	111.60
Endosulfan I	100	88	25.056	47.99	47.99	52.21	-27.18	123.16	-2.12	98.10
a-Chlordane	100	90	17.359	84.19	84.19	20.62	32.11	136.26	49.47	118.91
Dieldrin	100	88	14.129	86.75	86.75	16.29	44.36	129.14	58.49	115.01
4,4'-DDE	100	86	14.552	88.84	88.84	16.38	45.18	132.49	59.73	117.94
Endrin	100	90	16.003	103.93	103.93	15.40	55.93	151.94	71.93	135.94
Endosulfan II	100	90	26.333	55.56	55.56	47.40	-23.44	134.55	2.89	108.22
4,4'-DDD	100	70	16.822	92.19	92.19	18.25	41.72	142.65	58.54	125.83
Endrin Aldehyde	100	66	15.895	58.06	58.06	27.38	10.38	105.74	26.27	89.85
Endosulfan Sulfate	100	86	15.875	89.62	89.62	17.71	41.99	137.24	57.87	121.37
4,4'-DDT	100	86	26.110	98.49	98.49	26.51	20.16	176.82	46.27	150.71
Endrin Ketone	100	90	17.122	88.09	88.09	19.44	36.72	139.45	53.85	122.33
HBB	100	90	18.782	79.33	79.33	23.67	22.99	135.68	41.77	116.90
Methoxychlor	100	78	15.921	95.42	95.42	16.68	47.66	143.19	63.58	127.26
Mirex	100	90	13.867	83.69	83.69	16.57	42.09	125.29	55.96	111.42
BP-6	100	90	17.980	91.82	91.82	19.58	37.88	145.76	55.86	127.78
Aroclor 1016	500	56	74.193	439.36	87.87	16.89	43.36	132.39	58.19	117.55
Aroclor 1260	500	56	80.276	458.71	91.74	17.50	43.58	139.91	59.63	123.85

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8081/8082
MS/MSD - Soil

			PRECISION*			RPD		
Compound		PAIRS	SD of RPDs	Average RPD		UCL		UWL
a-BHC		45	13.819	13.783		41.46		27.64
b-BHC		45	9.845	9.998		29.54		19.69
g-BHC		45	13.896	12.560		41.69		27.79
d-BHC		44	28.112	25.210		84.34		56.22
Heptachlor		43	10.610	11.065		31.83		21.22
Aldrin		45	9.891	11.088		29.67		19.78
Hept-Epox.		45	9.470	9.773		28.41		18.94
g-Chlordane		45	9.406	9.668		28.22		18.81
Endosulfan I		44	36.962	28.489		110.89		73.92
a-Chlordane		45	25.342	13.555		76.02		50.68
Dieldrin		44	9.528	10.177		28.59		19.06
4,4'-DDE		43	10.348	10.026		31.05		20.70
Endrin		45	10.224	9.745		30.67		20.45
Endosulfan II		45	34.778	25.919		104.33		69.56
4,4'-DDD		35	15.039	11.143		45.12		30.08
Endrin Aldehyde		33	13.758	12.954		41.27		27.52
Endosulfan Sulfate		43	12.731	11.979		38.19		25.46
4,4'-DDT		43	11.645	10.944		34.94		23.29
Endrin Ketone		45	11.063	9.792		33.19		22.13
HBB		45	11.683	12.970		35.05		23.37
Methoxychlor		39	10.955	11.600		32.87		21.91
Mirex		45	10.434	9.791		31.30		20.87
BP-6		45	11.067	10.451		33.20		22.13
Aroclor 1016		28	12.119	12.422		36.36		24.24
Aroclor 1260		28	10.446	12.843		31.34		20.89

***Precision is from in batch MS/MSD spike duplicates**

MS= Matrix Spike

MSD= Matrix Spike Duplicate

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

SD= Standard Deviation

RPD= Relative Percent Difference

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method TO-15 AIR

		PRECISION*				RPD		
Compound	**PAIRS**	**SD of RPDs**	**Average RPD**			**UCL**		**UWL**
Dichlorodifluoromethane	16	3.328	3.622			9.98		6.66
Chloromethane	16	2.972	3.959			8.92		5.94
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	#	#	#			#		#
1,3-Butadiene	12	3.364	5.101			10.09		6.73
Vinyl Chloride	#	#	#			#		#
Bromomethane	#	#	#			#		#
Chloroethane	#	#	#			#		#
Acetonitrile	#	#	#			#		#
Trichlorofluoromethane	16	1.658	2.160			4.97		3.32
Acrylonitrile	#	#	#			#		#
1,1-Dichloroethylene	#	#	#			#		#
Methylene Chloride	10	2.737	3.516			8.21		5.47
1,1,2-Trichloro-1,2,2-Trifluoroethane	#	#	#			#		#
trans-1,2-Dichloroethylene	#	#	#			#		#
1,1-Dichloroethane	#	#	#			#		#
Methyl Tertiary Butyl Ether	#	#	#			#		#
Methyl Ethyl Ketone	#	#	#			#		#
2-Chloro-1,3-Butadiene	#	#	#			#		#
cis-1,2-Dichloroethylene	#	#	#			#		#
Hexane	#	#	#			#		#
Chloroform	#	#	#			#		#
1,2-Dichloroethane	#	#	#			#		#
1,1,1-Trichloroethane	#	#	#			#		#
Benzene	14	2.196	3.051			6.59		4.39
Carbon Tetrachloride	#	#	#			#		#
1,2-Dichloropropane	#	#	#			#		#
Bromodichloromethane	#	#	#			#		#
Trichloroethylene	#	#	#			#		#
2,2,4-Trimethylpentane	4	2.822	3.742			8.47		5.64
cis-1,3-Dichloropropylene	#	#	#			#		#
Methyl Isobutyl Ketone	#	#	#			#		#
trans-1,3-Dichloropropylene	#	#	#			#		#
1,1,2-Trichloroethane	#	#	#			#		#
Toluene	15	3.648	5.474			10.95		7.30
Dibromochloromethane	#	#	#			#		#
1,2-Dibromoethane	#	#	#			#		#
Tetrachloroethylene	#	#	#			#		#
Chlorobenzene	#	#	#			#		#
Ethylbenzene	3	4.308	5.975			12.93		8.62
m&p-Xylene	14	5.100	6.135			15.30		10.20
Bromoform	#	#	#			#		#
Styrene	#	#	#			#		#
1,1,2,2-Tetrachloroethane	#	#	#			#		#
o-Xylene	7	3.619	5.560			10.86		7.24
1,3,5-Trimethylbenzene	#	#	#			#		#
1,2,4-Trimethylbenzene	8	5.840	6.686			17.52		11.68
Benzyl Chloride	#	#	#			#		#
1,3-Dichlorobenzene	#	#	#			#		#
1,4-Dichlorobenzene	#	#	#			#		#
1,2-Dichlorobenzene	#	#	#			#		#
1,2,4-Trichlorobenzene	#	#	#			#		#
Hexachloro-1,3-Butadiene	#	#	#			#		#

SD= Standard Deviation
RPD= Relative Percent Difference

*** Precisision is from duplicate injections of the same canister.**
Units are in ppbv. **# Not enough data points**

UCL= Upper Control Limit
UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method TO-11
AIR

			ACCURACY*				Control Windows			
			Standard	Average	Recovery		% Recovery			
Compound	TV (mg/L)	N	Deviation (mg/L)	mg/L	%	% RSD	LCL	UCL	LWL	UWL
Formaldehyde	6.99	15	0.397	6.63	94.86	5.99	77.81	111.91	83.49	106.23
Acetaldehyde	5.10	15	0.272	4.34	85.07	6.27	69.08	101.07	74.41	95.73
Acetone	4.10	14	0.452	3.65	88.94	12.38	55.90	121.99	66.91	110.97
Propionaldehyde	4.10	15	0.304	3.63	88.56	8.38	66.31	110.81	73.73	103.40
Crotonaldehyde	3.57	15	0.347	2.77	77.70	12.51	48.54	106.85	58.26	97.13
n-Butyraldehyde	3.50	15	0.318	3.22	92.04	9.86	64.82	119.25	73.89	110.18
Benzaldehyde	2.70	15	0.248	2.58	95.45	9.63	67.86	123.04	77.06	113.85
Isovaleraldehyde	3.10	15	0.181	2.86	92.32	6.31	74.83	109.80	80.66	103.97
Valeraldehyde	3.10	15	0.168	3.12	100.55	5.40	84.25	116.85	89.68	111.42
o-Tolualdehyde	2.50	15	0.208	2.17	86.90	9.57	61.95	111.86	70.27	103.54
m,p-Tolualdehyde	5.00	15	0.449	4.61	92.19	9.73	65.27	119.10	74.24	110.13
Hexanaldehyde	2.80	15	0.180	2.32	82.99	7.76	63.68	102.30	70.12	95.86
2,5-Dimethylbenzaldehyde	2.34	15	0.184	1.98	84.41	9.32	60.81	108.00	68.68	100.14

*Accuracy is determined from spiking underivitized standard onto a blank cartridge.

		PRECISION**			RPD		
Compound		PAIRS	SD of RPDs	Average RPD		UCL	UWL
Formaldehyde		17	1.873	1.523		5.62	3.75
Acetaldehyde		17	2.003	1.783		6.01	4.01
Acetone		17	0.882	1.309		2.64	1.76
Propionaldehyde		12	4.904	4.974		14.71	9.81
Crotonaldehyde		1	#	#		#	#
n-Butyraldehyde		8	3.220	2.786		9.66	6.44
Benzaldehyde		3	#	#		#	#
Isovaleraldehyde		4	#	#		#	#
Valeraldehyde		4	#	#		#	#
o-Tolualdehyde		1	#	#		#	#
m,p-Tolualdehyde		1	#	#		#	#
Hexanaldehyde		5	#	#		#	#
2,5-Dimethylbenzaldehyde		0	#	#		#	#

**Precision is from duplicate injections of the same sample extract.

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

SD= Standard Deviation

RPD= Relative Percent Difference

Units are mg/L which are direct readings from the HPLC of the ester compound.

Insufficient data points

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
FULL LIST MS/MSD - Water

			ACCURACY				Control Windows			
							% Recovery			
Compound	TV (ug/L)	N	Standard Deviation (ug/L)	Average ug/L	Recovery %	% RSD	LCL	UCL	LWL	UWL
Dichlorodifluoromethane	50	8	5.309	53.79	107.58	9.87	75.73	139.43	86.34	128.81
Chloromethane	50	8	7.608	49.52	99.03	15.36	53.38	144.68	68.60	129.46
Vinyl Chloride	50	8	6.436	51.57	103.13	12.48	64.51	141.75	77.39	128.87
Bromomethane	50	8	11.744	45.30	90.60	25.92	20.14	161.06	43.63	137.58
Chloroethane	50	8	5.447	49.93	99.85	10.91	67.17	132.53	78.06	121.64
Trichlorofluoromethane	50	8	5.239	50.46	100.91	10.38	69.48	132.35	79.96	121.87
2-Propanone (acetone)	50	8	5.853	28.35	56.69	20.65	21.57	91.81	33.28	80.10
Diethylether	50	8	6.344	50.16	100.32	12.65	62.25	138.38	74.94	125.69
1,1-Dichloroethylene	50	8	5.788	50.70	101.41	11.42	66.68	136.14	78.26	124.56
Methyl Iodide	50	8	9.032	61.50	123.01	14.68	68.82	177.20	86.88	159.14
Acrylonitrile	50	8	7.254	51.05	102.09	14.21	58.57	145.62	73.08	131.11
Methylene Chloride	50	8	7.106	48.93	97.87	14.52	55.23	140.50	69.45	126.29
Carbon Disulfide	50	8	5.607	49.84	99.68	11.25	66.03	133.32	77.25	122.11
trans-1,2-Dichloroethylene	50	8	5.327	48.55	97.10	10.97	65.14	129.06	75.79	118.41
Methyl Tertiary Butyl Ether	50	8	5.935	49.48	98.95	12.00	63.34	134.56	75.21	122.69
1,1-Dichloroethane	50	8	5.437	47.55	95.10	11.43	62.48	127.72	73.35	116.85
2-Butanone (MEK)	50	8	4.415	37.40	74.80	11.81	48.31	101.29	57.14	92.46
cis-1,2-Dichloroethylene	50	8	5.223	47.08	94.16	11.09	62.83	125.50	73.27	115.06
Chloroform	50	8	4.822	47.69	95.37	10.11	66.44	124.31	76.08	114.66
Tetrahydrofuran	50	8	7.714	48.01	96.01	16.07	49.72	142.30	65.15	126.87
Bromochloromethane	50	8	4.951	48.47	96.94	10.21	67.24	126.65	77.14	116.75
1,1,1-Trichloroethane	50	8	4.486	48.15	96.29	9.32	69.37	123.21	78.34	114.24
1,2-Dichloroethane	50	8	4.671	49.00	97.99	9.53	69.97	126.02	79.31	116.68
Benzene	50	8	4.689	48.36	96.71	9.70	68.58	124.85	77.96	115.47
Carbon Tetrachloride	50	8	5.192	47.95	95.91	10.83	64.75	127.06	75.14	116.67
1,2-Dichloropropane	50	8	4.697	48.57	97.15	9.67	68.97	125.32	78.36	115.93
Trichloroethylene	50	8	4.753	46.22	92.43	10.28	63.91	120.95	73.42	111.44
Dibromomethane	50	8	4.898	49.23	98.45	9.95	69.06	127.84	78.86	118.04
Bromodichloromethane	50	8	4.702	48.88	97.75	9.62	69.54	125.97	78.95	116.56
4-Methyl-2-Pentanone (MIBK)	50	8	6.536	49.67	99.33	13.16	60.12	138.54	73.19	125.47

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
FULL LIST MS/MSD - Water

			ACCURACY				Control Windows			
							% Recovery			
Compound	TV (ug/L)	N	Standard Deviation (ug/L)	Average ug/L	Recovery %	% RSD	LCL	UCL	LWL	UWL
cis-1,3-Dichloropropene	50	8	4.928	48.05	96.10	10.26	66.53	125.67	76.39	115.81
trans-1,3-Dichloropropene	50	8	4.884	48.47	96.94	10.08	67.64	126.24	77.40	116.47
Toluene	50	8	4.815	47.16	94.31	10.21	65.43	123.20	75.05	113.57
1,1,2-Trichloroethane	50	8	5.105	48.65	97.30	10.49	66.67	127.93	76.88	117.72
2-Hexanone	50	8	4.489	39.50	78.99	11.36	52.06	105.92	61.04	96.95
Dibromochloromethane	50	8	5.130	49.29	98.59	10.41	67.81	129.37	78.07	119.11
1,2-Dibromoethane	50	8	5.436	49.10	98.20	11.07	65.58	130.81	76.45	119.94
Tetrachloroethylene	50	8	5.359	45.12	90.24	11.88	58.08	122.39	68.80	111.67
Chlorobenzene	50	8	5.664	46.08	92.16	12.29	58.18	126.15	69.50	114.82
1,1,1,2-Tetrachloroethane	50	8	4.769	49.17	98.34	9.70	69.72	126.95	79.26	117.41
Ethylbenzene	50	8	5.645	46.74	93.49	12.08	59.62	127.36	70.91	116.07
m & P-Xylene	50	8	4.834	44.65	89.30	10.83	60.30	118.31	69.97	108.64
Bromoform	50	8	7.080	47.21	94.42	15.00	51.94	136.91	66.10	122.75
Styrene	50	8	10.036	44.63	89.26	22.49	29.04	149.47	49.11	129.40
o-Xylene	50	8	5.589	46.86	93.72	11.93	60.18	127.25	71.36	116.07
1,1,2,2-Tetrachloroethane	50	8	5.833	48.98	97.96	11.91	62.96	132.96	74.62	121.29
1,2,3-Trichloropropane	50	8	6.771	50.33	100.66	13.45	60.03	141.29	73.58	127.74
1,4-Dichloro-2-butene	50	8	7.626	45.18	90.36	16.88	44.60	136.11	59.85	120.86
Isopropylbenzene	50	8	7.406	46.40	92.80	15.96	48.37	137.24	63.18	122.43
Bromobenzene	50	8	7.067	43.91	87.83	16.09	45.43	130.23	59.56	116.10
n-Propyl Benzene	50	8	8.381	45.35	90.69	18.48	40.41	140.98	57.17	124.22
1,3,5-Trimethylbenzene	50	8	7.842	44.88	89.76	17.47	42.71	136.81	58.40	121.13
tert-Butylbenzene	50	8	8.795	45.14	90.28	19.48	37.51	143.05	55.10	125.46
1,2,4-Trimethylbenzene	50	8	7.320	44.17	88.35	16.57	44.43	132.27	59.07	117.63
sec-Butylbenzene	50	8	10.069	43.36	86.72	23.22	26.31	147.13	46.45	127.00
1,3-Dichlorobenzene	50	8	9.290	43.38	86.75	21.42	31.01	142.49	49.59	123.91
1,4-Dichlorobenzene	50	8	8.829	42.61	85.21	20.72	32.24	138.18	49.90	120.53
p-isopropyl Toluene	50	8	10.072	43.11	86.22	23.36	25.78	146.65	45.93	126.50
1,2-Dichlorobenzene	50	8	9.561	43.00	86.00	22.24	28.63	143.36	47.75	124.24
n-Butylbenzene	50	8	11.442	41.81	83.63	27.37	14.97	152.28	37.86	129.40
Hexachloroethane	50	8	12.504	40.04	80.07	31.23	5.04	155.10	30.05	130.09
1,2-Dibromo-3-chloropropane	50	8	7.793	48.99	97.97	15.91	51.22	144.73	66.80	129.14
1,2,4-Trimethylbenzene	50	8	12.946	40.09	80.18	32.29	2.51	157.86	28.40	131.97
Naphthalene	50	8	12.330	44.20	88.40	27.90	14.42	162.38	39.08	137.72
1,2,3-Trichlorobenzene	50	8	12.793	39.72	79.44	32.21	2.68	156.20	28.26	130.61
2-Methyl Naphthalene	50	8	19.401	46.01	92.03	42.16	-24.38	208.43	14.42	169.63

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
FULL LIST MS/MSD - Water

		PRECISION*			RPD		
Compound		PAIRS	SD of RPDs	Average RPD		UCL	UWL
Dichlorodifluoromethane		4	7.787	8.04		23.36	15.57
Chloromethane		4	9.526	9.26		28.58	19.05
Vinyl Chloride		4	7.893	8.73		23.68	15.79
Bromomethane		4	14.895	17.68		44.68	29.79
Chloroethane		4	8.836	10.06		26.51	17.67
Trichlorofluoromethane		4	9.962	8.55		29.89	19.92
2-Propanone (acetone)		4	15.884	21.81		47.65	31.77
Diethylether		4	10.485	9.14		31.46	20.97
1,1-Dichloroethylene		4	10.219	9.02		30.66	20.44
Methyl Iodide		4	12.622	21.56		37.86	25.24
Acrylonitrile		4	11.757	10.19		35.27	23.51
Methylene Chloride		4	9.566	8.81		28.70	19.13
Carbon Disulfide		4	9.750	8.38		29.25	19.50
trans-1,2-Dichloroethylene		4	10.163	9.10		30.49	20.33
Methyl Tertiary Butyl Ether		4	10.019	10.80		30.06	20.04
1,1-Dichloroethane		4	9.622	9.04		28.87	19.24
2-Butanone (MEK)		4	11.506	11.36		34.52	23.01
cis-1,2-Dichloroethylene		4	10.187	9.30		30.56	20.37
Chloroform		4	9.669	9.04		29.01	19.34
Tetrahydrofuran		4	10.373	11.35		31.12	20.75
Bromochloromethane		4	10.302	9.61		30.91	20.60
1,1,1-Trichloroethane		4	10.484	9.42		31.45	20.97
1,2-Dichloroethane		4	10.401	8.98		31.20	20.80
Benzene		4	10.373	9.01		31.12	20.75
Carbon Tetrachloride		4	11.411	10.36		34.23	22.82
1,2-Dichloropropane		4	10.044	8.82		30.13	20.09
Trichloroethylene		4	11.073	9.41		33.22	22.15
Dibromomethane		4	9.912	9.71		29.74	19.82
Bromodichloromethane		4	10.386	9.34		31.16	20.77
4-Methyl-2-Pentanone (MIBK)		4	12.294	11.04		36.88	24.59
cis-1,3-Dichloropropene		4	10.759	9.78		32.28	21.52
trans-1,3-Dichloropropene		4	10.461	10.23		31.38	20.92
Toluene		4	11.380	10.03		34.14	22.76
1,1,2-Trichloroethane		4	10.080	9.34		30.24	20.16
2-Hexanone		4	12.465	11.10		37.40	24.93
Dibromochloromethane		4	9.298	10.98		27.89	18.60
1,2-Dibromoethane		4	10.271	10.43		30.81	20.54
Tetrachloroethylene		4	9.753	8.82		29.26	19.51
Chlorobenzene		4	9.285	9.52		27.86	18.57
1,1,1,2-Tetrachloroethane		4	9.617	9.82		28.85	19.23
Ethylbenzene		4	10.128	9.53		30.38	20.26
m & P-Xylene		4	10.118	9.39		30.35	20.24
Bromoform		4	9.097	12.64		27.29	18.19

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
FULL LIST MS/MSD - Water

		PRECISION*			RPD		
Compound		PAIRS	SD of RPDs	Average RPD		UCL	UWL
Styrene		4	8.762	11.40		26.29	17.52
o-Xylene		4	9.829	9.73		29.49	19.66
1,1,2,2-Tetrachloroethane		4	9.628	10.29		28.88	19.26
1,2,3-Trichloropropane		4	10.558	9.75		31.67	21.12
1,4-Dichloro-2-butene		4	12.220	11.96		36.66	24.44
Isopropylbenzene		4	10.335	9.73		31.01	20.67
Bromobenzene		4	9.588	10.77		28.76	19.18
n-Propyl Benzene		4	9.424	10.56		28.27	18.85
1,3,5-Trimethylbenzene		4	9.339	10.68		28.02	18.68
tert-Butylbenzene		4	10.023	10.85		30.07	20.05
1,2,4-Trimethylbenzene		4	10.267	11.03		30.80	20.53
sec-Butylbenzene		4	9.394	11.74		28.18	18.79
1,3-Dichlorobenzene		4	10.414	11.45		31.24	20.83
1,4-Dichlorobenzene		4	9.489	10.96		28.47	18.98
p-isopropyl Toluene		4	9.843	11.27		29.53	19.69
1,2-Dichlorobenzene		4	9.678	12.14		29.03	19.36
n-Butylbenzene		4	10.053	11.95		30.16	20.11
Hexachloroethane		4	10.547	14.11		31.64	21.09
1,2-Dibromo-3-chloropropane		4	12.279	12.88		36.84	24.56
1,2,4-Trimethylbenzene		4	9.808	14.66		29.42	19.62
Naphthalene		4	10.301	16.13		30.90	20.60
1,2,3-Trichlorobenzene		4	10.006	15.80		30.02	20.01
2-Methyl Naphthalene		4	10.296	23.36		30.89	20.59

*Precision is from in batch MS/MSD spike duplicates

MS= Matrix Spike

MSD= Matrix Spike Duplicate

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

SD= Standard Deviation

RPD= Relative Percent Difference

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260/624
Surrogate - Water
Mobile Lab

			ACCURACY				Control Windows			
				Average	Recovery		% Recovery			
Compound	TV (ug/L)	N	Standard Deviation (ug/L)	ug/L	%	% RSD	LCL	UCL	LWL	UWL
Dibromofluoromethane	50	1614	1.382	49.18	98.36	2.81	90.07	106.66	92.83	103.89
Toluene-d8	50	1666	0.825	50.35	100.69	1.64	95.74	105.64	97.39	103.99
4-Bromofluorobenzene	50	1666	0.854	50.22	100.45	1.70	95.32	105.57	97.03	103.87

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
MS/MSD - Water
Mobile Lab

ACCURACY

Compound	TV (ug/L)	N	Standard Deviation (ug/L)	Average ug/L	Recovery %	% RSD	Control Windows % Recovery LCL	UCL	LWL	UWL
1,1-Dichloroethylene	50	186	4.679	49.15	98.30	9.52	70.23	126.37	79.59	117.02
Benzene	50	186	2.169	51.92	103.85	4.18	90.83	116.86	95.17	112.52
Trichloroethylene	50	186	1.928	48.95	97.90	3.94	86.33	109.46	90.18	105.61
Toluene	50	186	2.037	51.27	102.54	3.97	90.32	114.76	94.39	110.69
Chlorobenzene	50	186	1.766	50.99	101.98	3.46	91.38	112.57	94.92	109.04

PRECISION*

Compound		PAIRS	SD of RPDs	Average RPD		RPD UCL		UWL
1,1-Dichloroethylene		93	2.015	2.23		6.05		4.03
Benzene		93	1.611	2.00		4.83		3.22
Trichloroethylene		93	1.644	2.04		4.93		3.29
Toluene		93	1.578	2.02		4.73		3.16
Chlorobenzene		93	1.668	1.93		5.01		3.34

***Precision is from in batch MS/MSD spike duplicates**

MS= Matrix Spike

MSD= Matrix Spike Duplicate

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

SD= Standard Deviation

RPD= Relative Percent Difference

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
Surrogate - Soil
Mobile Lab

			ACCURACY				Control Windows			
			Standard	Average	Recovery		% Recovery			
Compound	TV (ug/Kg)	N	Deviation (ug/Kg)	ug/Kg	%	% RSD	LCL	UCL	LWL	UWL
Dibromofluoromethane	2500	622	585.884	2291.35	91.65	25.57	21.35	161.96	44.78	138.52
Toluene-d8	2500	622	413.177	2336.26	93.45	17.69	43.87	143.03	60.40	126.50
4-Bromofluorobenzene	2500	622	653.249	2521.89	100.88	25.90	22.49	179.27	48.62	153.14

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
QA Summary for Year 2001
Method 8260
MS/MSD - Soil
Mobile Lab

ACCURACY

Compound	TV (ug/Kg)	N	Standard Deviation (ug/Kg)	Average Recovery ug/Kg	Average Recovery %	% RSD	Control Windows % Recovery LCL	UCL	LWL	UWL
1,1-Dichloroethylene	2500	84	248.051	2488.75	99.55	9.97	69.78	129.32	79.71	119.39
Benzene	2500	84	149.346	2624.09	104.96	5.69	87.04	122.89	93.02	116.91
Trichloroethylene	2500	84	109.781	2528.98	101.16	4.34	87.99	114.33	92.38	109.94
Toluene	2500	84	196.609	2501.09	100.04	7.86	76.45	123.64	84.31	115.77
Chlorobenzene	2500	84	101.555	2572.67	102.91	3.95	90.72	115.09	94.78	111.03

PRECISION*

Compound		PAIRS	SD of RPDs	Average RPD		RPD UCL		RPD UWL
1,1-Dichloroethylene		42	1.763	2.30		5.29		3.53
Benzene		42	1.298	1.77		3.89		2.60
Trichloroethylene		42	1.631	2.06		4.89		3.26
Toluene		42	1.545	1.75		4.64		3.09
Chlorobenzene		42	1.370	1.83		4.11		2.74

***Precision is from in batch MS/MSD spike duplicates**

MS= Matrix Spike

MSD= Matrix Spike Duplicate

TV= True Value

N= Number of data points

RSD= Relative Standard Deviation

SD= Standard Deviation

RPD= Relative Percent Difference

LCL= Lower Control Limit

UCL= Upper Control Limit

LWL= Lower Warning Limit

UWL= Upper Warning Limit

MDEQ Environmental Laboratory

INORGANIC UNIT 2001 AC AND PC SUMMARY

			ACCURACY					PRECISION *		
ELEMENT	EPA NO.	MATRIX	CONC (ug/l)	%RECOVERY MEAN	%RECOVERY S.D.	%RECOVERY RSD	N	MC (ug/l) MEAN	MC (ug/l) S.D.	N
Aluminum	200.8/6020	water	50	95.5	13.4	14.0	300	48.5	5.6	371
Antimony	200.8/6020	water	50	114.4	6.9	6.0	374	52	6.3	371
Arsenic	200.8/6020	water	50	101.3	8	7.9	373	50	3.8	371
Barium	6010	TCLP	100	91.6	6.03	6.6	14	185.2	10.9	10
Barium	200.8/6020	water	50	96.8	7.3	7.5	372	49.7	5.2	371
Beryllium	200.8/6020	water	50	103.4	10.7	10.3	373	50.3	3.9	371
Boron	200.7/6010	water	100	101.8	4.95	4.9	159	102.6	4.51	225
Cadmium	6010	TCLP	100	88.7	2.12	2.4	9	163.1	5.52	7
Cadmium	200.8/6020	water	50	100.4	4.5	4.5	379	50.2	3.3	371
Chromium	6010	TCLP	500	94.3	5.4	5.7	11	234.3	8.88	8
Chromium	7190	water	50	100.3	6.17	6.2	206	40.4	1.22	18
Chromium	200.7/6010	water	50	99.5	6.7	6.7	13	50.1	3.38	13
Cobalt	200.8/6020	water	50	95.8	6.8	7.1	378	50	3.8	371
Copper	6010	TCLP	500	93.6	3.2	3.4	8	324.8	3.9	5
Copper (63)	200.8/6020	water	50	92.4	7	7.6	373	50.6	4.4	371
Copper (65)	200.8/6020	water	50	92.5	6.6	7.1	372	50.6	4.4	371
Hex Chrom.	7196	water	361	95.8	3.9	4.1	92	No Hexavalent Cr present		
Iron	200.7/6010	water	500	99.4	4.3	4.3	188	506.5	17.8	274
Lead	6010	TCLP	1000	95.3	6.3	6.6	11	460.6	18.8	7
Lead	200.8/6020	water	50	100.6	8.4	8.4	377	50.9	3.6	371
Lithium	200.7/6010	water	100	99.8	4.31	4.3	148	99.4	7.45	204
Manganese	200.7/6010	water	50	101.5	4.9	4.9	21	51.2	1.92	29
Manganese	200.8/6020	water	50	97.8	7.9	8.1	312	50	3.6	371
Mercury	7470	water	4	99.7	2.8	2.8	196	1.27	0.042	20
Low Level Mercury	1631	water	0.005	101.9	6.6	6.5	9	5.23	0.39	18
Low Level Mercury	1631	water	0.003	94.9	8.8	9.3	50	5.97	0.91	20
Molybdenum	200.8/6020	water	50	104.9	7.2	6.9	374	53	4.2	371
Nickel	6010	TCLP	500	88	11.5	13.1	6	316.8	1.3	4
Nickel	200.8/6020	water	50	93.4	7.6	8.1	374	49.9	3.9	371
Selenium	7740	water	50	98.7	5.3	5.4	206	22.9	0.92	23
Silver	200.8/6020	water	50	89	9.7	10.90	365	51.1	4.6	371
Strontium	200.8/6020	water	50	98	9.1	9.29	304	50.7	5.2	371
Thallium	200.8/6020	water	50	99.1	8.8	8.88	377	51	6.4	371
Titanium	200.8/6020	water	50	100	8.2	8.20	376	50.9	4.3	371
Vanadium	200.8/6020	water	50	101	7.2	7.13	378	50.2	3.5	371
Zinc	6010	TCLP	100	100.1	6.9	6.9	12	90.4	5	9
Zinc	200.8/6020	water	50	97.2	9.2	9.46	327	49.9	3.4	371

*Precision is based on samples of known concentration and indicates between run precision.

MDEQ Environmental Laboratory

INORGANIC UNIT 2001 AC AND PC SUMMARY

			ACCURACY					PRECISION *		
	EPA		CONC	%RECOVERY			N	MC (mg/l)		N
ELEMENT	NO.	MATRIX	(mg/l)	MEAN	S.D.	RSD		MEAN	S.D.	
Alkalinity #	310.2	water	100	93.2	4.05	4.3	179	251	5.15	47
Calcium	7140	water	30	101	6.11	6.1	202	26.6	0.69	107
Chloride #	375.2	water	100	99.3	2.11	2.1	236	50.4	0.51	64
Magnesium	7450	water	10	101.6	7.52	7.4	209	7.36	0.12	108
Oil & Grease	413.1	water	40	88	7.95	9.0	18	35.1	2.99	18
Potassium	7610	water	1.6	102.8	7.44	7.2	173	1.05	0.02	90
Sodium	7770	water	30	99.9	5.94	6.0	187	10.5	0.47	94
Sulfate #	325.2	water	100	99.6	2.93	2.9	224	49.4	1.27	67

*Precision is based on samples of known concentration and indicates between run precision.
#Precision is based on a blank spiked with a known conc./between run precision.

MDEQ Environmental Laboratory

INORGANIC UNIT 2001 AC AND PC SUMMARY

			ACCURACY					PRECISION *		
ELEMENT	EPA NO.	MATRIX	CONC (ug/l)	%RECOVERY MEAN	%RECOVERY S.D.	%RECOVERY RSD	N	NIST REF.(mg/kg) MEAN	NIST REF.(mg/kg) S.D.	N
Aluminum	6010	sediment	2500	103.8	3.47	3.3	61	8912	477	21
Arsenic	7061	sediment	40	97.1	10.1	10.3	30	15.7	0.33	16
Arsenic	7060	sediment	5	92.3	7.7	8.3	87	6.73	0.42	41
Barium	6010	sediment	100	97.5	4.6	4.7	100	100.9	4.9	52
Beryllium	6010	sediment	50	97.6	3.4	3.5	41	0.64	0.037	22
Cadmium	7130	sediment	100	96.9	3.4	3.5	111	4.9	0.28	59
Chromium	6010	sediment	100	96.4	4.7	4.9	113	58.6	2.1	58
Cobalt	7200	sediment	500	94.2	4.3	4.6	45	8.4	0.4	24
Copper	7210	sediment	500	95.8	3.4	3.6	114	75.9	3.6	56
Iron	7380	sediment	2500	100.8	8.09	5.0	40	19300	1018	24
Lead	7420	sediment	500	95.6	3.3	3.5	113	121	8.6	52
Lithium	7430	sediment	100	97.3	2.9	3.0	38	16.3	0.5	22
Manganese	7460	sediment	100	98.6	5.4	5.5	44	355.3	10.4	26
Mercury	7471	sediment	4	99.8	5.3	5.3	101	1.42	0.063	36
Molybdenum	6010	sediment	100	92	5.5	6.0	49	2.1	0.6	25
Nickel	7520	sediment	500	94.8	4.4	4.6	111	34.9	1.4	58
Selenium	7741	sediment	40	91.1	9.4	10.3	32	1.36	0.08	17
Selenium	7740	sediment	5	90.8	6.42	7.1	70	1.13	0.16	34
Silver	7761	sediment	2	95.4	6.63	7.0	98	0.99	0.14	34
Strontium	6010	sediment	100	98	3.2	3.3	42	69.2	1.8	21
Thallium	7841	sediment	5	94.2	6.45	6.9	14	1.62	0.18	6
Titanium	6010	sediment	250	99.7	2.85	2.9	58	140.2	18.6	17
Vanadium	6010	sediment	100	95.4	3.3	3.5	37	21.7	0.95	18
Zinc	7950	sediment	500	95.5	3.77	4.0	112	258	8.9	56

*Precision is calculated from reference standards and indicates between run precision.

MDEQ Environmental Laboratory

INORGANIC UNIT 2001 AC AND PC SUMMARY

			ACCURACY					PRECISION *		
	EPA		CONC	% RECOVERY			N	OIL REF. (mg/kg)		N
ELEMENT	NO.	MATRIX	(ug/l)	MEAN	S.D.	RSD		MEAN	S.D.	
Arsenic	7061	organic	40	87.3	9.05	10.4	9	90.5	6.28	8
Barium	6010	organic	20	106	8.49	8.0	10	93.3	4.9	11
Cadmium	7130	organic	20	95	3.53	3.7	10	92.2	4.7	11
Copper	7210	organic	100	102.3	4.9	1.9	10	97.8	3.9	11
Chromium	6010	organic	20	100.5	6.47	6.4	10	100.3	3.5	11
Lead	7420	organic	100	95	4.9	5.2	10	95.5	3.5	11
Mercury	7471	organic	4	85.6	14.9	17.5	5	66.01	10.45	7
Nickel	7520	organic	20	101	4.46	4.4	10	97.7	3.8	11
Zinc	7950	organic	100	96.7	5.6	5.8	8	90.4	4	10

*Precision is calculated from reference standards and indicates between run precision.

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

LABORATORY SERVICES SECTION
ENVIRONMENTAL LABORATORY
QUALITY ASSURANCE PROGRAM PLAN

APPENDIX E

Drinking Water Laboratory Test List

QAPP-Ver2.0, Revison 0
May 5, 2003

TEST CODE | TEST FEE | METHOD INFORMATION - METALS LAB GROUP

CAG 16.00 SILVER BY ICP/MS [Ref.: EPA200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 5 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

CAL 16.00 ALUMINUM BY ICP/MS [Ref.: EPA200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 6 Rev. 07/18/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

ALUMINUM BY ICP/MS [# 001 Code: AL Rev: 07/18/96 R# 5]
PQL: 0.05 mg/L Min DL: 0.05 mg/L RMCL: NONE ESTABLISHED CAS: 7429-90-5

CAS 16.00 ARSENIC BY ICP/MS [Ref.: EPA200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 7 Rev. 04/15/97]

REPORTED ANALYTES FOR TEST PROCEDURE:

ARSENIC [# 002 Code: AS Rev: 04/15/97 R# 6]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: 0.01 mg/L REGULATORY MCL CAS: 7440-38-2

CB 16.00 BORON BY ICP/MS [Ref.: EPA 200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 8 Rev. 07/21/97]

REPORTED ANALYTES FOR TEST PROCEDURE:

CBA 16.00 BARIUM BY ICP/MS [Ref.: EPA200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 9 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

BARIUM [# 001 Code: BA Rev: 07/01/96 R# 7]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: 2 mg/L REGULATORY MCL CAS: 7440-39-3

CBE 16.00 BERYLLIUM BY ICP/MS [Ref.: EPA200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 10 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

BERYLLIUM [# 001 Code: BE Rev: 07/01/96 R# 8]
PQL: 0.0004 mg/L Min DL: 0.0004 mg/L RMCL: 0.004 mg/L REGULATORY MCL CAS: 7440-41-7

CCA 18.00 CALCIUM BY FLAME AA [Ref.: SM3111A-B]
Sample Kit: 32, 33 Holding Limit: 180 days Lab Group: METALS [Rec# 11 Rev. 04/15/97]

REPORTED ANALYTES FOR TEST PROCEDURE:

CALCIUM [# 002 Code: CA Rev: 04/15/97 R# 9]
PQL: 0.3 mg/L Min DL: 0.3 mg/L RMCL: NONE ESTABLISHED CAS: 7440-70-2

TEST CODE	TEST FEE	METHOD INFORMATION - METALS LAB GROUP
CCD	16.00	CADMIUM BY ICP/MS [Ref.: EPA200.8]
		Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 13 Rev. 07/01/96]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		CADMIUM [# 001 Code: CD Rev: 07/01/96 R# 10]
		PQL: 0.0003 mg/L Min DL: 0.0003 mg/L RMCL: 0.005 mg/L REGULATORY MCL CAS: 7440-43-9
CCR	16.00	CHROMIUM BY ICP/MS [Ref.: EPA200.8]
		Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 19 Rev. 07/01/96]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		CHROMIUM [# 001 Code: CR Rev: 07/01/96 R# 15]
		PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: 0.1 mg/L REGULATORY MCL CAS: 7440-47-3
CCU	6.00	COPPER BY ICP/MS [Ref.: EPA 200.8]
		Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 20 Rev. 11/01/01]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		COPPER [# 001 Code: CU Rev: 11/01/01 R# 16]
		PQL: 0.05 mg/L Min DL: 0.05 mg/L RMCL: NONE ESTABLISHED CAS: 7440-50-8
CFE	5.00	IRON BY ICP/MS [Ref.: EPA 200.8]
		Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 25 Rev. 11/01/01]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		IRON [# 001 Code: FE Rev: 11/01/01 R# 24]
		PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: NONE ESTABLISHED CAS: 7439-89-6
CHG	16.00	MERCURY TESTING BY ICP/MS [Ref.: EPA200.8]
		Sample Kit: 36ME Holding Limit: 28 days Lab Group: METALS [Rec# 27 Rev. 04/16/97]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		MERCURY [# 002 Code: HG Rev: 04/16/97 R# 26]
		PQL: 0.0001 mg/L Min DL: 0.0001 mg/L RMCL: 0.002 mg/L REGULATORY MCL CAS: 7439-97-6
CK	13.00	POTASSIUM BY FLAME AA [Ref.: SM3500KD]
		Sample Kit: 32, 33 Holding Limit: 180 days Lab Group: METALS [Rec# 29 Rev. 07/01/96]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		POTASSIUM [# 001 Code: K Rev: 07/01/96 R# 28]
		PQL: 0.03 mg/L Min DL: 0.03 mg/L RMCL: NONE ESTABLISHED CAS: 7440-09-7

TEST CODE	TEST FEE	METHOD INFORMATION - METALS LAB GROUP
CMAS	12.00	ARSENIC BY ICP/MS [Ref.: EPA200.8]
		Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 30 Rev. 05/05/97]
		REPORTED ANALYTES FOR TEST PROCEDURE:
CMCU	5.00	COPPER BY ICP/MS [Ref.: EPA 200.8]
		Sample Kit: 36CC Holding Limit: 180 days Lab Group: METALS [Rec# 31 Rev. 01/04/00]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		COPPER [# 001 Code: CU Rev: 01/04/00 R# 29]
		PQL: 0.05 mg/L Min DL: 0.05 mg/L RMCL: NONE ESTABLISHED CAS: 7440-50-8
CMG	5.00	MAGNESIUM BY FLAME AA [Ref.: SM3111A-B]
		Sample Kit: 32, 33 Holding Limit: 180 days Lab Group: METALS [Rec# 32 Rev. 04/16/97]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		MAGNESIUM [# 002 Code: MG Rev: 04/16/97 R# 30]
		PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: NONE ESTABLISHED CAS: 7439-95-4
CMN	4.00	MANGANESE BY ICP/MS [Ref.: EPA 200.8]
		Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 33 Rev. 11/01/01]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		MANGANESE [# 001 Code: MN Rev: 11/01/01 R# 31]
		PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: NONE ESTABLISHED CAS: 7439-96-5
CMPB	12.00	LEAD BY ICP/MS [Ref.: EPA200.8]
		Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 34 Rev. 05/05/97]
		REPORTED ANALYTES FOR TEST PROCEDURE:
CNA	5.00	SODIUM BY FLAME AA [Ref.: SM3500-NAD]
		Sample Kit: 32, 33 Holding Limit: 180 days Lab Group: METALS [Rec# 35 Rev. 04/16/97]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		SODIUM [# 002 Code: NA Rev: 04/16/97 R# 33]
		PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: NONE ESTABLISHED CAS: 7440-23-5
CNI	16.00	NICKEL BY ICP/MS [Ref.: EPA200.8]
		Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 37 Rev. 07/01/96]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		NICKEL [# 001 Code: NI Rev: 07/01/96 R# 35]
		PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: 0.1 mg/L REGULATORY MCL CAS: 7440-02-0

TEST CODE	TEST FEE	METHOD INFORMATION - METALS LAB GROUP

CPB 16.00 LEAD (TOTAL) BY ICP/MS [Ref.: EPA 200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 40 Rev. 12/10/99]

REPORTED ANALYTES FOR TEST PROCEDURE:

LEAD BY ICP/MS [# 001 Code: PB Rev: 12/10/99 R# 38]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 7439-92-1

CPM1 20.00 IRON/MANGANESE/COPPER/ZINC BY ICP/MS [Ref.: EPA 200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 42 Rev. 01/14/02]

REPORTED ANALYTES FOR TEST PROCEDURE:

COPPER [# 004 Code: CU Rev: 01/14/02 R# 40]
PQL: 0.05 mg/L Min DL: 0.05 mg/L RMCL: NONE ESTABLISHED CAS: 7440-50-8

IRON [# 003 Code: FE Rev: 01/14/02 R# 41]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: NONE ESTABLISHED CAS: 7439-89-6

MANGANESE [# 002 Code: MN Rev: 01/14/02 R# 42]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: NONE ESTABLISHED CAS: 7439-96-5

ZINC [# 001 Code: ZN Rev: 01/14/02 R# 43]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: NONE ESTABLISHED CAS: 7440-66-6

CPM2 18.00 CALCIUM/MAGNESIUM/SODIUM BY FLAME AA [Ref.: SM3111B, SM3500]
Sample Kit: 32, 33 Holding Limit: 180 days Lab Group: METALS [Rec# 43 Rev. 07/01/97]

REPORTED ANALYTES FOR TEST PROCEDURE:

CALCIUM [# 001 Code: CA Rev: 07/01/97 R# 44]
PQL: 0.3 mg/L Min DL: 0.3 mg/L RMCL: NONE ESTABLISHED CAS: 7440-70-2

MAGNESIUM [# 002 Code: MG Rev: 07/01/97 R# 45]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: NONE ESTABLISHED CAS: 7439-95-4

SODIUM [# 003 Code: NA Rev: 07/01/97 R# 46]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: NONE ESTABLISHED CAS: 7440-23-5

CSB 16.00 ANTIMONY BY ICP/MS [Ref.: EPA200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 48 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

ANTIMONY [# 001 Code: SB Rev: 07/01/96 R# 63]
PQL: 0.0006 mg/L Min DL: 0.0006 mg/L RMCL: 0.006 mg/L REGULATORY MCL CAS: 7440-36-0

TEST CODE	TEST FEE	METHOD INFORMATION - METALS LAB GROUP

CSE 16.00 SELENIUM BY ICP/MS [Ref.: EPA200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 49 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

SELENIUM [# 001 Code: SE Rev: 07/01/96 R# 64]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: 0.05 mg/L REGULATORY MCL CAS: 7782-49-2

CSN 16.00 TIN BY ICP/MS [Ref.: EPA200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 52 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

TIN [# 001 Code: SN Rev: 07/01/96 R# 67]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 7440-31-5

CSR 16.00 STRONTIUM BY ICP/MS [Ref.: EPA200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 53 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

STRONTIUM [# 001 Code: SR Rev: 07/01/96 R# 68]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: NONE ESTABLISHED CAS: 7440-24-6

CTL 16.00 THALLIUM BY ICP/MS [Ref.: EPA200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 56 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

THALLIUM [# 001 Code: TL Rev: 07/01/96 R# 71]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.002 mg/L REGULATORY MCL CAS: 7440-28-0

CTURA 0.00 Turbidity for Acidified Samples [Ref.: EPA180.1]
Sample Kit: 36ME 36C Holding Limit: NONE Lab Group: METALS [Rec# 58 Rev. 11/30/99]

REPORTED ANALYTES FOR TEST PROCEDURE:

TURBIDITY FOR ACIDIFIED SAMPLES [# 001 Code: CTURA Rev: 11/30/99 R# 73]
PQL: 1 NTU Min DL: 1 NTU RMCL: NONE ESTABLISHED CAS:

CU 16.00 URANIUM [Ref.: EPA200.8]
Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 59 Rev. 11/03/98]

REPORTED ANALYTES FOR TEST PROCEDURE:

URANIUM [# 001 Code: U Rev: 11/03/98 R# 74]
PQL: 0.0003 mg/L Min DL: 0.0003 mg/L RMCL: 0.03 mg/L REGULATORY MCL CAS: 7440-61-1

TEST CODE	TEST FEE	METHOD INFORMATION - METALS LAB GROUP
CU-235	16.00	URANIUM 235 [Ref.: EPA200.8]
		Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 60 Rev. 06/26/01]
		REPORTED ANALYTES FOR TEST PROCEDURE:
CU235	16.00	URANIUM 235 [Ref.: EPA200.8]
		Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 61 Rev. 05/10/01]
		REPORTED ANALYTES FOR TEST PROCEDURE:
CV	16.00	VANADIUM [Ref.: EPA 200.8]
		Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 62 Rev. 01/24/03]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		VANADIUM [# 001 Code: V Rev: 01/24/03 R# 75]
		PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 7440-62-2
CZN	5.00	ZINC BY ICP/MS [Ref.: EPA 200.8]
		Sample Kit: 36ME Holding Limit: 180 days Lab Group: METALS [Rec# 82 Rev. 11/01/01]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		ZINC [# 001 Code: ZN Rev: 11/01/01 R# 296]
		PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: NONE ESTABLISHED CAS: 7440-66-6

TEST CODE	TEST FEE	METHOD INFORMATION - INORG LAB GROUP

CCARB 10.00 CARBONATE AS CaCO3 BY TITRATION [Ref.: EPA310.1]
Sample Kit: 32 Holding Limit: ASAP Lab Group: INORG [Rec# 12 Rev. 05/09/97]

REPORTED ANALYTES FOR TEST PROCEDURE:

CCHS 30.00 HYDROGEN SULFIDE BY TITRATION [Ref.: SM4500-S-D]
Sample Kit: 36HS Holding Limit: 28 days Lab Group: INORG [Rec# 14 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

HYDROGEN SULFIDE (QUANT) [# 001 Code: CHS Rev: 07/01/96 R# 11]
PQL: 1 mg/L Min DL: 1 mg/L RMCL: NONE ESTABLISHED CAS: 7783-06-4

CCL2 0.00 RESIDUAL CHLORINE [Ref.:]
Sample Kit: 32 Holding Limit: ASAP Lab Group: INORG [Rec# 15 Rev. 05/13/97]

REPORTED ANALYTES FOR TEST PROCEDURE:

CCLO4 75.00 PERCHLORATE [Ref.: EPA 314.0]
Sample Kit: 32 Holding Limit: 28 days Lab Group: INORG [Rec# 16 Rev. 08/21/00]

REPORTED ANALYTES FOR TEST PROCEDURE:

PERCHLORATE [# 001 Code: CLO4 Rev: 08/21/00 R# 12]
PQL: 0.004 mg/L Min DL: 0.004 mg/L RMCL: NONE ESTABLISHED CAS: 14797-73-0

CCN 25.00 CYANIDE BY DISTILLATION/COLORIMETRY [Ref.: EPA335.2]
Sample Kit: 36CN(a) Holding Limit: 14 days Lab Group: INORG [Rec# 17 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

CYANIDE [# 001 Code: CN Rev: 07/01/96 R# 13]
PQL: 0.02 mg/L Min DL: 0.02 mg/L RMCL: 0.2 mg/L REGULATORY MCL CAS: 57-12-5

CCON 12.00 SPECIFIC CONDUCTANCE [Ref.: EPA120.1]
Sample Kit: 32,33 Holding Limit: 28 days Lab Group: INORG [Rec# 18 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

CONDUCTIVITY [# 001 Code: CON Rev: 07/01/96 R# 14]
PQL: 1 umhos Min DL: 1 umhos RMCL: NONE ESTABLISHED CAS: COND-00-C

CD 13.00 TBAS SCREENING [Ref.: IN-HOUSE]
Sample Kit: 32 Holding Limit: 24 days Lab Group: INORG [Rec# 21 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

TBAS SCREENING [# 001 Code: D Rev: 07/01/96 R# 17]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: NONE ESTABLISHED CAS: CD-00-C

TEST CODE	TEST FEE	METHOD INFORMATION - INORG LAB GROUP

CDBP 75.00 INORGANIC DISINFECTION BYPRODUCTS [Ref.: EPA 300.1]
Sample Kit: 32a Holding Limit: 14 days Lab Group: INORG [Rec# 22 Rev. 08/21/00]

REPORTED ANALYTES FOR TEST PROCEDURE:

BROMIDE [# 003 Code: BR Rev: 08/21/00 R# 18]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS:

BROMATE [# 002 Code: BRO3 Rev: 08/21/00 R# 19]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: 0.01 mg/L MCL CAS:

CHLORITE [# 001 Code: CLO2 Rev: 08/21/00 R# 20]
PQL: 0.006 mg/L Min DL: 0.006 mg/L RMCL: 1.0 mg/L MCL CAS:

CHLORATE [# 004 Code: CLO3 Rev: 08/21/00 R# 21]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: NONE ESTABLISHED CAS:

CFDY 17.00 FLUORESCENT DYE (PRESENCE/ABSENCE) [Ref.: MDEQ]
Sample Kit: 36FY Holding Limit: 24 days Lab Group: INORG [Rec# 24 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

FLUORESCENT DYE (QUAL.) [# 001 Code: FDY Rev: 07/01/96 R# 23]
PQL: Min DL: RMCL: NONE ESTABLISHED CAS: FDY-00-C

CH2S 23.00 HYDROGEN SULFIDE (PRESENCE/ABSENCE) [Ref.: IN-HOUSE]
Sample Kit: 32 Holding Limit: 24 days Lab Group: INORG [Rec# 26 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

HYDROGEN SULFIDE (QUAL.) [# 001 Code: H2S Rev: 07/01/96 R# 25]
PQL: Min DL: RMCL: NONE ESTABLISHED CAS: 7783-06-4

CNH3 30.00 AMMONIA BY SPECIFIC ION ELECTRODE [Ref.: SM4500NH3-F]
Sample Kit: 36AC Holding Limit: 28 days Lab Group: INORG [Rec# 36 Rev. 04/01/97]

REPORTED ANALYTES FOR TEST PROCEDURE:

AMMONIA as N [# 001 Code: NH3 Rev: 07/01/96 R# 34]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: NONE ESTABLISHED CAS: 7664-41-7

CPH 13.00 PH DETERMINATION [Ref.: EPA150.1]
Sample Kit: 32, 33 Holding Limit: ASAP Lab Group: INORG [Rec# 41 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

pH [# 001 Code: PH Rev: 07/01/96 R# 39]
PQL: 2.0 Min DL: 2.0 RMCL: NONE ESTABLISHED CAS: PH-00-C

TEST CODE	TEST FEE	METHOD INFORMATION - INORG LAB GROUP

CPO4 17.00 PHOSPHATE,ORTHO- TESTING BY COLORIMETRY [Ref.: SM4500P-E]
Sample Kit: 32, 33 Holding Limit: 2 days Lab Group: INORG [Rec# 44 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

ORTHO-PHOSPHATE AS P [# 001 Code: PO4 Rev: 07/01/96 R# 47]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: NONE ESTABLISHED CAS: 7664-38-2

CRA 14.00 AUTOMATED PARTIAL CHEMICAL BATTERY [Ref.: SM4500NO3H, SM3]
Sample Kit: 32 Holding Limit: 2 days Lab Group: INORG [Rec# 45 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

FLUORIDE (AUTOMATED) [# 005 Code: AF Rev: 07/01/96 R# 48]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: 4.0 mg/L REGULATORY MCL CAS: 16984-48-8

IRON (AUTOMATED) [# 007 Code: AFE Rev: 07/01/96 R# 49]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: NONE ESTABLISHED CAS: 7439-89-6

HARDNESS AS CaCO3 [# 006 Code: AHA Rev: 07/01/96 R# 50]
PQL: 10 mg/L Min DL: 10 mg/L RMCL: NONE ESTABLISHED CAS: HARD-00-C

SODIUM (AUTOMATED) [# 009 Code: ANA Rev: 07/01/96 R# 51]
PQL: 5 mg/L Min DL: 5 mg/L RMCL: NONE ESTABLISHED CAS: 7440-23-5

CHLORIDE (AUTOMATED) [# 004 Code: CL Rev: 07/01/96 R# 52]
PQL: 4 mg/L Min DL: 4 mg/L RMCL: NONE ESTABLISHED CAS: 7647-14-5

NITRITE AS N (AUTOMATED) [# 003 Code: NO2 Rev: 07/01/96 R# 53]
PQL: 0.05 mg/L Min DL: 0.05 mg/L RMCL: 1 mg/L REGULATORY MCL CAS: 14797-65-0

NITRATE AS N (AUTOMATED) [# 001 Code: NO3 Rev: 07/01/96 R# 54]
PQL: 0.4 mg/L Min DL: 0.4 mg/L RMCL: 10 mg/L REGULATORY MCL CAS: 14797-55-8

SULFATE (AUTOMATED) [# 008 Code: SO4 Rev: 07/01/96 R# 55]
PQL: 5 mg/L Min DL: 5 mg/L RMCL: NONE ESTABLISHED CAS: 14808-79-8

CRES 10.00 RESISTANCE FOR DEMINERALIZED WATER [Ref.: EPA 120.1]
Sample Kit: 32,33 Holding Limit: ASAP Lab Group: INORG [Rec# 46 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

RESISTANCE [# 001 Code: RES Rev: 07/01/96 R# 56]
PQL: 1 ohms Min DL: 1 ohms RMCL: NONE ESTABLISHED CAS: RES-00-C

TEST CODE	TEST FEE	METHOD INFORMATION - INORG LAB GROUP

CRM 14.00 AUTOMATED CHEMISTRY FOR COMPLETE MINERALS [Ref.: EPA335.2, SM350]
Sample Kit: 33 Holding Limit: 2 days Lab Group: INORG [Rec# 47 Rev. 05/05/97]

REPORTED ANALYTES FOR TEST PROCEDURE:

FLUORIDE (AUTOMATED) [# 004 Code: AF Rev: 05/05/97 R# 57]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: 4.0 mg/L REGULATORY MCL CAS: 16984-48-8

HARDNESS AS CaCO3 [# 005 Code: AHA Rev: 05/05/97 R# 58]
PQL: 10 mg/L Min DL: 10 mg/L RMCL: NONE ESTABLISHED CAS: HARD-00-C

CHLORIDE (AUTOMATED) [# 003 Code: CL Rev: 05/05/97 R# 59]
PQL: 4 mg/L Min DL: 4 mg/L RMCL: NONE ESTABLISHED CAS: 7647-14-5

NITRITE AS N (AUTOMATED) [# 002 Code: NO2 Rev: 05/05/97 R# 60]
PQL: 0.05 mg/L Min DL: 0.05 mg/L RMCL: 1 mg/L REGULATORY MCL CAS: 14797-65-0

NITRATE AS N (AUTOMATED) [# 001 Code: NO3 Rev: 05/05/97 R# 61]
PQL: 0.4 mg/L Min DL: 0.4 mg/L RMCL: 10 mg/L REGULATORY MCL CAS: 14797-55-8

SULFATE (AUTOMATED) [# 006 Code: SO4 Rev: 05/05/97 R# 62]
PQL: 5 mg/L Min DL: 5 mg/L RMCL: NONE ESTABLISHED CAS: 14808-79-8

CSI 14.00 SILICA AS SIO2 BY COLORIMETRY [Ref.: SM4500SIO2-C]
Sample Kit: 32,33 Holding Limit: 28 days Lab Group: INORG [Rec# 50 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

SILICA AS SiO2 [# 001 Code: SI Rev: 07/01/96 R# 65]
PQL: 0.2 mg/L Min DL: 0.2 mg/L RMCL: NONE ESTABLISHED CAS: 7440-21-3

CTALK 16.00 TOTAL ALKALINITY AS CaCO3 BY TITRATION [Ref.: SM2320 B]
Sample Kit: 32, 33 Holding Limit: 14 days Lab Group: INORG [Rec# 54 Rev. 05/15/97]

REPORTED ANALYTES FOR TEST PROCEDURE:

TOTAL ALKALINITY AS CaCO3 [# 001 Code: TOTAL AL Rev: 05/15/97 R# 69]
PQL: 1 mg/L Min DL: 1 mg/L RMCL: NONE ESTABLISHED CAS: TALK-00-C

CTAN 11.00 TANNATES (PRESENCE/ABSENCE) [Ref.: IN-HOUSE]
Sample Kit: 32 Holding Limit: ASAP Lab Group: INORG [Rec# 55 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

TANNATES(QUAL) [# 001 Code: TAN Rev: 07/01/96 R# 70]
PQL: Min DL: RMCL: NONE ESTABLISHED CAS: 1401-55-4

TEST CODE	TEST FEE	METHOD INFORMATION - INORG LAB GROUP
CTOC	30.00	TOTAL ORGANIC CARBON [Ref.: SM 5310C]
		Sample Kit: 36TO Holding Limit: 28 days Lab Group: INORG [Rec# 57 Rev. 08/22/00]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		TOTAL ORGANIC CARBON [# 001 Code: TOC Rev: 08/22/00 R# 72]
		PQL: 0.5 mg/L Min DL: 0.5 mg/L RMCL: NONE ESTABLISHED CAS: TOC-00-C

TEST CODE	TEST FEE	METHOD INFORMATION - VOC LAB GROUP

CXEV 70.00 EDB AND DBCP BY ECD [Ref.: EPA504.1]
Sample Kit: 36VO Holding Limit: 28 days Lab Group: VOC [Rec# 68 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

DIBROMO-3-CHLOROPROPANE, 1,2- [# 002 Code: DBCP Rev: 07/01/96 R# 95]
PQL: 0.00005 mg/L Min DL: 0.00005 mg/L RMCL: 0.0002 mg/L REGULATORY MCL
CAS: 96-12-8

DIBROMOETHANE, 1,2 [# 001 Code: EDB Rev: 07/01/96 R# 96]
PQL: 0.00005 mg/L Min DL: 0.00005 mg/L RMCL: 0.00005 mg/L REGULATORY MCL
CAS: 106-93-4

CXMP 55.00 MAX POTENTIAL TTHM FORMATION BY GC/MS [Ref.: EPA524.2]
Sample Kit: 36VOc Holding Limit: 7 days Lab Group: VOC [Rec# 74 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

MPF CHLORODIBROMOMETHANE [# 003 Code: CHBR2CL Rev: 07/01/96 R# 133]
PQL: 0.0004 mg/L Min DL: 0.0004 mg/L RMCL: 0.1000 mg/L REGULATORY MCL
CAS: 124-48-1

MPF BROMOFORM [# 004 Code: CHBR3 Rev: 07/01/96 R# 134]
PQL: 0.0004 mg/L Min DL: 0.0004 mg/L RMCL: 0.1000 mg/L REGULATORY MCL
CAS: 75-25-2

MPF DICHLOROBROMOMETHANE [# 002 Code: CHBRCL2 Rev: 07/01/96 R# 135]
PQL: 0.0004 mg/L Min DL: 0.0004 mg/L RMCL: 0.1000 mg/L REGULATORY MCL
CAS: 75-27-4

MPF CHLOROFORM [# 001 Code: CHCL3 Rev: 07/01/96 R# 136]
PQL: 0.0004 mg/L Min DL: 0.0004 mg/L RMCL: 0.1000 mg/L REGULATORY MCL
CAS: 67-66-3

MAX POTENTIAL TRIHALOMETHANES [# 005 Code: TTHM Rev: 07/01/96 R# 137]
PQL: 0.0004 mg/L Min DL: 0.0004 mg/L RMCL: 0.1000 mg/L REGULATORY MCL
CAS: MPTHM-00-C

CXPD 115.00 P-DIOXANE BY ITD - MAGNUM [Ref.: IN-HOUSE]
Sample Kit: 36VO Holding Limit: 14 days Lab Group: VOC [Rec# 77 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

DIOXANE,1,4- [# 001 Code: PDIOXANE Rev: 07/01/96 R# 160]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 123-91-1

CXTM 55.00 TOTAL TRIHALOMETHANE MONITORING [Ref.: EPA524.2]
Sample Kit: 36VO Holding Limit: 14 days Lab Group: VOC [Rec# 80 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

CHLORODIBROMOMETHANE [# 003 Code: CHBR2CL Rev: 07/01/96 R# 228]

PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 0.080 mg/L REGULATORY MCL CAS: 124-48-1

BROMOFORM [# 004 Code: CHBR3 Rev: 07/01/96 R# 229]

PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 0.080 mg/L REGULATORY MCL CAS: 75-25-2

TEST CODE | TEST FEE | METHOD INFORMATION - VOC | LAB GROUP

CXTM (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

DICHLOROBROMOMETHANE [# 002 Code: CHBRCL2 Rev: 07/01/96 R# 230]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 0.080 mg/L REGULATORY MCL CAS: 75-27-4

CHLOROFORM [# 001 Code: CHCL3 Rev: 07/01/96 R# 231]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 0.080 mg/L REGULATORY MCL CAS: 67-66-3

TOTAL TRIHALOMETHANES [# 005 Code: TTHM Rev: 07/01/96 R# 232]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 0.080 mg/L REGULATORY MCL CAS: TTHM-00-C

CXVO 90.00 ORGANIC SOLVENTS BY GC/MS [Ref.: EPA524.2]
Sample Kit: 36VO Holding Limit: 14 days Lab Group: VOC [Rec# 81 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

BROMOBENZENE [# 039 Code: BRBZ Rev: 07/01/96 R# 233]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 108-86-1

BENZENE [# 038 Code: BZ Rev: 07/01/96 R# 234]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 0.005 mg/L REGULATORY MCL CAS: 71-43-2

DICHLOROETHYLENE,1,2-CIS [# 016 Code: C12DCE Rev: 07/01/96 R# 235]
PQL: 0.0004 mg/L Min DL: 0.0002 mg/L RMCL: 0.07 mg/L REGULATORY MCL CAS: 156-59-2

DICHLOROPROPENE,1,3-CIS [# 035 Code: C13DCPE Rev: 07/01/96 R# 236]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 10061-01-5

HEXACHLOROBUTADIENE [# 024 Code: C46 Rev: 07/01/96 R# 237]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 87-68-3

CARBON TETRACHLORIDE [# 004 Code: CCL4 Rev: 07/01/96 R# 238]
PQL: 0.0004 mg/L Min DL: 0.0003 mg/L RMCL: 0.005 mg/L REGULATORY MCL CAS: 56-23-5

DICHLORODIFLUOROMETHANE [# 012 Code: CF2CL2 Rev: 07/01/96 R# 239]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 75-71-8

FLUOROTRICHLOROMETHANE [# 023 Code: CFCL3 Rev: 07/01/96 R# 240]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 75-69-4

DIBROMOMETHANE [# 010 Code: CH2BR2 Rev: 07/01/96 R# 241]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 74-95-3

BROMOCHLOROMETHANE [# 001 Code: CH2BRCL Rev: 07/01/96 R# 242]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 74-97-5

TEST CODE | TEST FEE | METHOD INFORMATION - VOC | LAB GROUP

CXVO (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

METHYLENE CHLORIDE [# 026 Code: CH2CL2 Rev: 07/01/96 R# 243]
PQL: 0.0006 mg/L Min DL: 0.0003 mg/L RMCL: 0.005 mg/L REGULATORY MCL CAS: 75-09-2

BROMOMETHANE [# 003 Code: CH3BR Rev: 07/01/96 R# 244]
PQL: 0.001 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 74-83-9

CHLORODIBROMOMETHANE [# 006 Code: CHBR2CL Rev: 07/01/96 R# 245]
PQL: 0.0005 mg/L Min DL: 0.0002 mg/L RMCL: 0.080 mg/L REGULATORY MCL CAS: 124-48-1

BROMOFORM [# 002 Code: CHBR3 Rev: 07/01/96 R# 246]
PQL: 0.0005 mg/L Min DL: 0.0002 mg/L RMCL: 0.080 mg/L REGULATORY MCL CAS: 75-25-2

DICHLOROBROMOMETHANE [# 011 Code: CHBRCL2 Rev: 07/01/96 R# 247]
PQL: 0.0005 mg/L Min DL: 0.0002 mg/L RMCL: 0.080 mg/L REGULATORY MCL CAS: 75-27-4

CHLOROFORM [# 008 Code: CHCL3 Rev: 07/01/96 R# 248]
PQL: 0.0005 mg/L Min DL: 0.0002 mg/L RMCL: 0.080 mg/L REGULATORY MCL CAS: 67-66-3

CHLOROBENZENE [# 005 Code: CLBZ Rev: 07/01/96 R# 249]
PQL: 0.0005 mg/L Min DL: 0.0003 mg/L RMCL: 0.1 mg/L REGULATORY MCL CAS: 108-90-7

CHLOROTOLUENE (COMBINED) [# 043 Code: CLTOL4 Rev: 07/01/96 R# 250]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 100-44-7

DICHLOROBENZENE,1,2- [# 048 Code: DCBZ12 Rev: 07/01/96 R# 251]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 0.6 mg/L REGULATORY MCL CAS: 95-50-1

DICHLOROBENZENE,1,3- [# 047 Code: DCBZ13 Rev: 07/01/96 R# 252]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 541-73-1

DICHLOROBENZENE,1,4- [# 046 Code: DCBZ14 Rev: 07/01/96 R# 253]
PQL: 0.0004 mg/L Min DL: 0.0003 mg/L RMCL: 0.075 mg/L REGULATORY MCL CAS: 106-46-7

DICHLOROETHYLENE,1,1- [# 015 Code: DCE11 Rev: 07/01/96 R# 254]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 0.007 mg/L REGULATORY MCL CAS: 75-35-4

DICHLOROETHANE,1,1- [# 013 Code: DCEA11 Rev: 07/01/96 R# 255]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 75-34-3

DICHLOROETHANE,1,2- [# 014 Code: DCEA12 Rev: 07/01/96 R# 256]
PQL: 0.0005 mg/L Min DL: 0.0002 mg/L RMCL: 0.005 mg/L REGULATORY MCL CAS: 107-06-2

DICHLOROPROPANE,1,2- [# 018 Code: DCPA12 Rev: 07/01/96 R# 257]
PQL: 0.0004 mg/L Min DL: 0.0002 mg/L RMCL: 0.005 mg/L REGULATORY MCL CAS: 78-87-5

TEST CODE | TEST FEE | METHOD INFORMATION - VOC | LAB GROUP

CXVO (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

DICHLOROPROPANE,1,3- [# 019 Code: DCPA13 Rev: 07/01/96 R# 258]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 142-28-9

DICHLOROPROPANE,2,2- [# 020 Code: DCPA22 Rev: 07/01/96 R# 259]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 590-20-7

DICHLOROPROPENE,1,1- [# 021 Code: DCPE11 Rev: 07/01/96 R# 260]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 563-58-6

ETHYLBENZENE [# 049 Code: ETBZ Rev: 07/01/96 R# 261]
PQL: 0.0005 mg/L Min DL: 0.0002 mg/L RMCL: 0.7 mg/L REGULATORY MCL CAS: 100-41-4

CHLOROETHANE [# 007 Code: ETCL Rev: 07/01/96 R# 262]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 75-00-3

ISOPROPYL BENZENE [# 053 Code: IPRBZ Rev: 07/01/96 R# 263]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 98-82-8

ISOPROPYL TOLUENE, PARA- [# 057 Code: IPRTOL Rev: 07/01/96 R# 264]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 99-87-6

CHLOROMETHANE [# 009 Code: MECL Rev: 07/01/96 R# 265]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 74-87-3

METHYL ETHYL KETONE [# 050 Code: MEK Rev: 07/01/96 R# 266]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 78-93-3

METHYL ISOBUTYL KETONE [# 051 Code: MIBK Rev: 07/01/96 R# 267]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 108-10-1

METHYL TERT-BUTYL ETHER [# 065 Code: MTBE Rev: 07/01/96 R# 268]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 1634-04-4

NAPHTHALENE [# 052 Code: NAPH Rev: 07/01/96 R# 269]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 91-20-3

BUTYLBENZENE, NORMAL- [# 042 Code: NBUBZ Rev: 07/01/96 R# 270]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 104-51-8

NITROBENZENE [# 067 Code: NBZ Rev: 07/01/96 R# 271]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: NONE ESTABLISHED CAS: 98-95-3

PROPYLBENZENE, NORMAL- [# 054 Code: NPRBZ Rev: 07/01/96 R# 272]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 103-65-1

TEST CODE | TEST FEE | METHOD INFORMATION - VOC | LAB GROUP

CXVO (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

TETRACHLOROETHYLENE [# 029 Code: PCE Rev: 07/01/96 R# 273]
PQL: 0.0005 mg/L Min DL: 0.0003 mg/L RMCL: 0.005 mg/L REGULATORY MCL CAS: 127-18-4

BUTYLBENZENE, SEC- [# 041 Code: SBUBZ Rev: 07/01/96 R# 274]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 135-98-8

STYRENE [# 055 Code: STYRN Rev: 07/01/96 R# 275]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 0.1 mg/L REGULATORY MCL CAS: 100-42-5

DICHLOROETHYLENE,1,2-TRANS [# 017 Code: T12DCE Rev: 07/01/96 R# 276]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 0.1 mg/L REGULATORY MCL CAS: 156-60-5

DICHLOROPROPENE,1,3-TRANS [# 036 Code: T13DCPE Rev: 07/01/96 R# 277]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 10061-02-6

BUTYLBENZENE, TERT- [# 040 Code: TBUBZ Rev: 07/01/96 R# 278]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 98-06-6

TRICHLOROBENZENE,1,2,3- [# 059 Code: TCBZ123 Rev: 07/01/96 R# 279]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 87-61-6

TRICHLOROBENZENE,1,2,4- [# 058 Code: TCBZ124 Rev: 07/01/96 R# 280]
PQL: 0.0005 mg/L Min DL: 0.0003 mg/L RMCL: 0.07 mg/L REGULATORY MCL CAS: 120-82-1

TRICHLOROETHYLENE [# 033 Code: TCE Rev: 07/01/96 R# 281]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 0.005 mg/L REGULATORY MCL CAS: 79-01-6

TETRACHLOROETHANE,1,1,1,2- [# 027 Code: TCE1112 Rev: 07/01/96 R# 282]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 630-20-6

TETRACHLOROETHANE,1,1,2,2- [# 028 Code: TCE1122 Rev: 07/01/96 R# 283]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 79-34-5

TRICHLOROETHANE,1,1,1- [# 031 Code: TCEA111 Rev: 07/01/96 R# 284]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 0.2 mg/L REGULATORY MCL CAS: 71-55-6

TRICHLOROETHANE,1,1,2- [# 032 Code: TCEA112 Rev: 07/01/96 R# 285]
PQL: 0.0005 mg/L Min DL: 0.0004 mg/L RMCL: 0.005 mg/L REGULATORY MCL CAS: 79-00-5

TRICHLOROPROPANE,1,2,3- [# 034 Code: TCPA123 Rev: 07/01/96 R# 286]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 96-18-4

TETRAHYDROFURAN [# 066 Code: THF Rev: 07/01/96 R# 287]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 109-99-9

TEST CODE | TEST FEE | METHOD INFORMATION - VOC | LAB GROUP

CXVO (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

TRIMETHYLBENZENE,1,2,4- [# 060 Code: TMBZ124 Rev: 07/01/96 R# 288]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 95-63-6

TRIMETHYLBENZENE,1,3,5- [# 061 Code: TMBZ135 Rev: 07/01/96 R# 289]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 108-67-8

TOLUENE [# 056 Code: TOL Rev: 07/01/96 R# 290]
PQL: 0.0005 mg/L Min DL: 0.0002 mg/L RMCL: 1 mg/L REGULATORY MCL CAS: 108-88-3

TOTAL TRIHALOMETHANES [# 030 Code: TTHM Rev: 07/01/96 R# 291]
PQL: 0.0005 mg/L Min DL: 0.0002 mg/L RMCL: 0.080 mg/L REGULATORY MCL CAS: TTHM-00-C

VINYL CHLORIDE [# 037 Code: VC Rev: 07/01/96 R# 292]
PQL: 0.0004 mg/L Min DL: 0.0004 mg/L RMCL: 0.002 mg/L REGULATORY MCL CAS: 75-01-4

XYLENES (TOTAL) [# 064 Code: XYLENS Rev: 07/01/96 R# 293]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 10 mg/L REGULATORY MCL CAS: 1330-20-7

XYLENE,META-&PARA- [# 062 Code: XYLMP Rev: 07/01/96 R# 294]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 10 mg/L REGULATORY MCL CAS: 1330-20-7

XYLENE, ORTHO- [# 063 Code: XYLO Rev: 07/01/96 R# 295]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: 10 mg/L REGULATORY MCL CAS: 95-47-6

TEST CODE	TEST FEE	METHOD INFORMATION - SOC LAB GROUP

CDCPA 95.00 TOTAL DCPA, mono and di-acid degradates [Ref.: EPA 515.4]
Sample Kit: 36NV Holding Limit: 14 days Lab Group: SOC [Rec# 23 Rev. 06/05/01]

REPORTED ANALYTES FOR TEST PROCEDURE:

TOTAL DCPA, MONO & DI-ACID DEGRADATES [# 001 Code: DCPA Rev: 06/05/01 R# 22]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 1861-32-1

CXAC 150.00 ACIDIC PHENOLS BY ITD [Ref.: IN-HOUSE]
Sample Kit: 36NV Holding Limit: 7 days Lab Group: SOC [Rec# 63 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

CHLORO-M-CRESOL,4- [# 001 Code: CLMCRL4 Rev: 07/01/96 R# 76]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 1321-10-4

CHLOROPHENOL,2- [# 002 Code: CLPHL2 Rev: 07/01/96 R# 77]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 95-57-8

CHLOROPHENOL,3- [# 003 Code: CLPHL3 Rev: 07/01/96 R# 78]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 108-43-0

DICHLOROPHENOL,3,5- [# 007 Code: DCLPHL35 Rev: 07/01/96 R# 79]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 591-35-5

DICHLOROPHENOL,2,4- [# 004 Code: DCPHL24 Rev: 07/01/96 R# 80]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 120-83-2

DICHLOROPHENOL,2,5- [# 005 Code: DCPHL25 Rev: 07/01/96 R# 81]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 583-78-8

DICHLOROPHENOL,3,4- [# 006 Code: DCPHL34 Rev: 07/01/96 R# 82]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 95-77-2

DIMETHYLPHENOL,2,3- [# 008 Code: DMPHL23 Rev: 07/01/96 R# 83]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 526-75-0

DIMETHYLPHENOL,2,4- [# 009 Code: DMPHL24 Rev: 07/01/96 R# 84]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 105-67-9

DIMETHYLPHENOL,2,5- [# 010 Code: DMPHL25 Rev: 07/01/96 R# 85]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 95-87-4

ETHYLPHENOL [# 013 Code: ETPHL Rev: 07/01/96 R# 86]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 90-00-6

ISOPHORONE [# 014 Code: IPHOR Rev: 07/01/96 R# 87]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 78-59-1

NITRO-P-CRESOL,2- [# 015 Code: NOPCRL2 Rev: 07/01/96 R# 88]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 119-33-5

TEST CODE | TEST FEE | METHOD INFORMATION - SOC LAB GROUP

CXAC (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

NITROPHENOL,2- [# 016 Code: NOPHL2 Rev: 07/01/96 R# 89]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 88-75-5

NITROPHENOL,4- [# 017 Code: NOPHL4 Rev: 07/01/96 R# 90]
PQL: 0.02 mg/L Min DL: 0.02 mg/L RMCL: NONE ESTABLISHED CAS: 100-02-7

PHENOL [# 019 Code: PHL Rev: 07/01/96 R# 91]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 108-95-2

TRICHLOROPHENOL,2,4,6- [# 020 Code: TCP246 Rev: 07/01/96 R# 92]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 88-06-2

CXBN 80.00 AROMATIC AMINES BY ECD [Ref.: IN-HOUSE]
Sample Kit: 36NV Holding Limit: 7 days Lab Group: SOC [Rec# 64 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

CXDQ 150.00 DIQUAT BY HPLC [Ref.: EPA549.2]
Sample Kit: 36DQ Holding Limit: 7 days Lab Group: SOC [Rec# 65 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

DIQUAT [# 001 Code: DIQUAT Rev: 07/01/96 R# 93]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: 0.02 mg/L REGULATORY MCL CAS: 2764-72-9

CXEN 150.00 ENDOTHALL BY GC/ITD [Ref.: EPA548.1]
Sample Kit: 36EN Holding Limit: 7 days Lab Group: SOC [Rec# 66 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

ENDOTHALL [# 001 Code: ENDOTHAL Rev: 07/01/96 R# 94]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: 0.1 mg/L REGULATORY MCL CAS: 129-67-9

CXET 80.00 HALOGENATED ETHERS BY ITD [Ref.: IN-HOUSE]
Sample Kit: 36NV Holding Limit: 7 days Lab Group: SOC [Rec# 67 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

TEST CODE	TEST FEE	METHOD INFORMATION - SOC LAB GROUP
CXGY	100.00	GLYPHOSATE BY HPLC/PCD [Ref.: EPA547]
		Sample Kit: 36GY Holding Limit: 14 days Lab Group: SOC [Rec# 69 Rev. 07/01/96]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		GLYPHOSATE [# 001 Code: GLYFOSAT Rev: 07/01/96 R# 97]
		PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: 0.7 mg/L REGULATORY MCL CAS: 1071-83-6
CXHA	120.00	DALAPON & HALOACETIC ACIDS BY GC/ECD [Ref.: EPA552.1]
		Sample Kit: 36HA Holding Limit: 28 days Lab Group: SOC [Rec# 70 Rev. 01/14/02]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		BROMOACETIC ACID [# 002 Code: BAA Rev: 01/14/02 R# 98]
		PQL: 0.004 mg/L Min DL: 0.004 mg/L RMCL: 0.06 mg/L MCL CAS: 79-08-3
		BROMOCHLOROACETIC ACID [# 007 Code: BCAA Rev: 01/14/02 R# 99]
		PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 5589-96-3
		CHLOROACETIC ACID [# 001 Code: CAA Rev: 01/14/02 R# 100]
		PQL: 0.004 mg/L Min DL: 0.004 mg/L RMCL: NONE ESTABLISHED CAS: 79-11-8
		DALAPON [# 006 Code: DALAPON Rev: 01/14/02 R# 101]
		PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: 0.2 mg/L REGULATORY MCL CAS: 75-99-0
		DIBROMOACETIC ACID [# 005 Code: DBAA Rev: 01/14/02 R# 102]
		PQL: 0.001 mg/L Min DL: 0.0005 mg/L RMCL: 0.06 mg/L MCL CAS: 631-64-1
		DICHLOROACETIC ACID [# 003 Code: DCAA Rev: 01/14/02 R# 103]
		PQL: 0.002 mg/L Min DL: 0.001 mg/L RMCL: 0.06 mg/L MCL CAS: 79-43-6
		TRICHLOROACETIC ACID [# 004 Code: TCAA Rev: 01/14/02 R# 104]
		PQL: 0.001 mg/L Min DL: 0.0004 mg/L RMCL: 0.06 mg/L MCL CAS: 76-03-9
		TOTAL HALOACETIC ACIDS (FIVE) [# 008 Code: THA Rev: 01/14/02 R# 105]
		PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: 0.06 mg/L MCL CAS:
CXHB	95.00	CHLORINATED ACID HERBICIDES BY GC/ECD [Ref.: EPA515.2]
		Sample Kit: 36NV Holding Limit: 14 days Lab Group: SOC [Rec# 71 Rev. 07/01/96]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		2,4,5-T [# 003 Code: 245T Rev: 07/01/96 R# 106]
		PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 93-76-5
		2,4,5-TP (SILVEX) [# 005 Code: 245TP Rev: 07/01/96 R# 107]
		PQL: 0.0003 mg/L Min DL: 0.0003 mg/L RMCL: 0.05 mg/L REGULATORY MCL CAS: 93-72-1
		2,4-D [# 008 Code: 24D Rev: 07/01/96 R# 108]
		PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: 0.07 mg/L REGULATORY MCL CAS: 94-75-7

TEST CODE | TEST FEE | METHOD INFORMATION - SOC LAB GROUP

CXHB (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

ACIFLUORFEN [# 006 Code: ACFLO Rev: 07/01/96 R# 109]
PQL: 0.004 mg/L Min DL: 0.004 mg/L RMCL: NONE ESTABLISHED CAS: 50594-66-6

BENTAZON [# 002 Code: BENTAZON Rev: 07/01/96 R# 110]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 25057-89-0

DCPA, MONO [# 010 Code: DACTHAL Rev: 07/01/96 R# 111]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 867-54-7

DICAMBA [# 007 Code: DICAMBA Rev: 07/01/96 R# 112]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 1918-00-9

DINOSEB [# 004 Code: DINOSEB Rev: 07/01/96 R# 113]
PQL: 0.0003 mg/L Min DL: 0.0003 mg/L RMCL: 0.007 mg/L REGULATORY MCL CAS: 88-85-7

PENTACHLOROPHENOL [# 009 Code: PCP Rev: 07/01/96 R# 114]
PQL: 0.00006 mg/L Min DL: 0.00006 mg/L RMCL: 0.001 mg/L REGULATORY MCL CAS: 87-86-5

PICLORAM [# 001 Code: PICLORAM Rev: 07/01/96 R# 115]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: 0.5 mg/L REGULATORY MCL CAS: 1918-02-1

CXLP 95.00 CARBAMATES BY HPLC/PCD [Ref.: EPA531.2]
Sample Kit: 36LP(a) Holding Limit: 28 days Lab Group: SOC [Rec# 72 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

ALDICARB [# 001 Code: ALDICARB Rev: 07/01/96 R# 116]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 116-06-3

ALDICARB SULFOXIDE [# 003 Code: ALDSFOXD Rev: 07/01/96 R# 117]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 1646-87-3

ALDICARB SULFONE [# 002 Code: ALDSUFON Rev: 07/01/96 R# 118]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 1646-88-4

HYDROXYCARBOFURAN, 3- [# 010 Code: CARB3OH Rev: 07/01/96 R# 119]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 16655-82-6

CARBARYL [# 005 Code: CARBARYL Rev: 07/01/96 R# 120]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 63-25-2

CARBOFURAN [# 006 Code: CARBFURN Rev: 07/01/96 R# 121]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: 0.04 mg/L REGULATORY MCL CAS: 1563-66-2

TEST CODE | TEST FEE | METHOD INFORMATION - SOC | LAB GROUP

CXLP (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

METHIOCARB [# 007 Code: METHCARB Rev: 07/01/96 R# 122]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 2032-65-7

METHOMYL [# 008 Code: METHOMYL Rev: 07/01/96 R# 123]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 16752-77-5

OXAMYL [# 009 Code: OXAMYL Rev: 07/01/96 R# 124]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: 0.2 mg/L REGULATORY MCL CAS: 23135-22-0

PROPOXUR (BAYGON) [# 004 Code: PROPOXUR Rev: 07/01/96 R# 125]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 114-26-1

CXLU 110.00 UV ACTIVE PESTICIDES BY HPLC/UV [Ref.: IN-HOUSE]
Sample Kit: 36NV Holding Limit: 28 days Lab Group: SOC [Rec# 73 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

BARBAN [# 001 Code: BARBAN Rev: 07/01/96 R# 126]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 101-27-9

DIURON [# 003 Code: DIURON Rev: 07/01/96 R# 127]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 330-54-1

FLUOMETURON [# 004 Code: FLUMTRON Rev: 07/01/96 R# 128]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 2164-17-2

LINURON [# 005 Code: LINURON Rev: 07/01/96 R# 129]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 330-55-2

NEBURON [# 007 Code: NEBURON Rev: 07/01/96 R# 130]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 555-37-3

PROPANIL [# 008 Code: PROPANIL Rev: 07/01/96 R# 131]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 709-98-8

PROPHAM [# 009 Code: PROPHAM Rev: 07/01/96 R# 132]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 122-42-9

TEST CODE	TEST FEE	METHOD INFORMATION - SOC LAB GROUP

CXNP 80.00 NITROGEN CONTAINING PESTICIDES BY GC/MS [Ref.: EPA525.2]
Sample Kit: 36NV Holding Limit: 7 days Lab Group: SOC [Rec# 75 Rev. 05/01/97]

REPORTED ANALYTES FOR TEST PROCEDURE:

CXPA 110.00 AROMATICS BY GC/MS [Ref.: EPA525.2]
Sample Kit: 36NV Holding Limit: 7 days Lab Group: SOC [Rec# 76 Rev. 01/12/00]

REPORTED ANALYTES FOR TEST PROCEDURE:

ACENAPHTHENE [# 001 Code: ACNPHEN Rev: 01/12/00 R# 138]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 83-32-9

ACENAPHTHYLENE [# 002 Code: ACNPHYN Rev: 01/12/00 R# 139]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 208-96-8

ANTHRACENE [# 003 Code: ANTH Rev: 01/12/00 R# 140]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 120-12-7

BENZO(A)ANTHRACENE [# 004 Code: BZAANTH Rev: 01/12/00 R# 141]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 56-55-3

BENZO(A)PYRENE [# 005 Code: BZAPYR Rev: 01/12/00 R# 142]
PQL: 0.00006 mg/L Min DL: 0.00006 mg/L RMCL: 0.0002 mg/L REGULATORY MCL
CAS: 50-32-8

BENZO(b)FLUORANTHENE [# 006 Code: BZBFLRN Rev: 01/12/00 R# 143]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 205-82-3

BENZO(g,h,i)PERYLENE [# 007 Code: BZGHIPR Rev: 01/12/00 R# 144]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 191-24-2

BENZO(k)FLUORANTHENE [# 008 Code: BZKFLRN Rev: 01/12/00 R# 145]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 207-08-9

CHRYSENE [# 011 Code: CHRYSN Rev: 01/12/00 R# 146]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 218-01-9

CHLORONAPHTHALENE, 2- [# 010 Code: CLNAPH2 Rev: 01/12/00 R# 147]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 91-58-7

DIBENZ(a,h)ANTHRACENE [# 012 Code: DBZANTH Rev: 01/12/00 R# 148]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 53-70-3

DI(ETHYLHEXYL)ADIPATE [# 029 Code: DEHADI Rev: 01/12/00 R# 149]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: 0.4 mg/L REGULATORY MCL CAS: 103-23-1

BIS(2-ETHYLHEXYL)PHTHALATE [# 013 Code: DEHP Rev: 01/12/00 R# 150]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: 0.006 mg/L REGULATORY MCL CAS: 117-81-7

DINITROTOLUENE, 2, 4 [# 019 Code: DNTOL24 Rev: 01/12/00 R# 151]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 121-14-2

TEST CODE | TEST FEE | METHOD INFORMATION - SOC | LAB GROUP

CXPA (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

DINITROTOLUENE, 2, 6- [# 018 Code: DNTOL26 Rev: 01/12/00 R# 152]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 606-20-2

FLUORENE [# 022 Code: FLRENE Rev: 01/12/00 R# 153]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 86-73-7

FLUORANTHENE [# 021 Code: FLRN Rev: 01/12/00 R# 154]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 206-44-0

INDENO(1,2,3-CD)PYRENE [# 023 Code: INDENO Rev: 01/12/00 R# 155]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 193-39-5

METHYLNAPHTHALENE,1- [# 024 Code: MENAPH1 Rev: 07/01/96 R# 156]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 90-12-0

METHYLNAPHTHALENE,2- [# 025 Code: MENAPH2 Rev: 07/01/96 R# 157]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 91-57-6

PHENANTHRENE [# 027 Code: PHENAN Rev: 01/12/00 R# 158]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 85-01-8

PYRENE [# 028 Code: PYR Rev: 01/12/00 R# 159]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 129-00-0

CXPO 150.00 ORGANOPHOSPHATE PESTICIDES BY GC/MS [Ref.: IN-HOUSE]
Sample Kit: 36NV Holding Limit: NONE Lab Group: SOC [Rec# 78 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

CHLORPYRIFOS [# 012 Code: CHLRPYRF Rev: 07/01/96 R# 161]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 05598-15-2

DIAZINON [# 001 Code: DAZNON Rev: 07/01/96 R# 162]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 333-41-5

DIMETHOATE [# 011 Code: DIMETATE Rev: 07/01/96 R# 163]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 60-51-5

DISULFOTON [# 002 Code: DSULFTON Rev: 07/01/96 R# 164]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 298-04-4

ETHION [# 004 Code: ETHION Rev: 07/01/96 R# 165]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 563-12-2

TEST CODE | TEST FEE | METHOD INFORMATION - SOC | LAB GROUP

CXPO (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

ETHYL PARATHION [# 005 Code: ETPTHON Rev: 07/01/96 R# 166]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 56-38-2

FENAMINPHOS [# 010 Code: FNMNPHOS Rev: 07/01/96 R# 167]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 22224-92-6

FONOFOS [# 003 Code: FONFOS Rev: 07/01/96 R# 168]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 944-22-9

AZINOPHOS,METHYL- [# 006 Code: GUTHION Rev: 07/01/96 R# 169]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 86-50-0

METHYL PARATHION [# 007 Code: MEPTHON Rev: 07/01/96 R# 170]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 298-00-0

MALATHION [# 008 Code: MLTHON Rev: 07/01/96 R# 171]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 121-75-5

CARBOPHENOTHION (TRITHION) [# 009 Code: TRITHON Rev: 07/01/96 R# 172]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 786-19-6

CXPT 120.00 PESTICIDES SCREENING BY ECD & NPD [Ref.: EPA525.2]
Sample Kit: 36NV Holding Limit: 7 days Lab Group: SOC [Rec# 79 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

ACETOCHLOR [# 057 Code: ACTCHLOR Rev: 07/01/96 R# 173]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 34256-82-1

ALACHLOR [# 037 Code: ALACHLOR Rev: 07/01/96 R# 174]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.002 mg/L REGULATORY MCL CAS: 15972-60-8

ALDRIN [# 004 Code: ALDRIN Rev: 07/01/96 R# 175]
PQL: 0.0004 mg/L Min DL: 0.0004 mg/L RMCL: NONE ESTABLISHED CAS: 309-00-2

AMETRYN [# 036 Code: AMETRYN Rev: 07/01/96 R# 176]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 834-12-8

ATRAZINE [# 038 Code: ATRAZINE Rev: 07/01/96 R# 177]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.003 mg/L REGULATORY MCL CAS: 1912-24-9

HEXACHLOROCYCLOHEXANE (ALPHA-BHC) [# 005 Code: BHC1 Rev: 07/01/96 R# 178]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 319-84-6

TEST CODE | TEST FEE | METHOD INFORMATION - SOC | LAB GROUP

CXPT (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

HEXACHLOROCYCLOHEXANE (BETA-BHC) [# 006 Code: BHC2 Rev: 07/01/96 R# 179]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 319-85-7

LINDANE (GAMMA-BHC) [# 002 Code: BHC3 Rev: 07/01/96 R# 180]
PQL: 0.00004 mg/L Min DL: 0.00004 mg/L RMCL: 0.0002 mg/L REGULATORY MCL
CAS: 58-89-9

HEXACHLOROCYCLOHEXANE (DELTA-BHC) [# 007 Code: BHC4 Rev: 07/01/96 R# 181]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 319-86-8

BROMACIL [# 032 Code: BROMACIL Rev: 07/01/96 R# 182]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 314-40-9

BUTACHLOR [# 051 Code: BUTACHLO Rev: 07/01/96 R# 183]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 23184-66-9

BUTYLATE [# 040 Code: BUTYLATE Rev: 07/01/96 R# 184]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 2008-41-5

HEXACHLOROCYCLOPENTADIENE [# 020 Code: C56 Rev: 07/01/96 R# 185]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.05 mg/L REGULATORY MCL CAS: 77-47-4

OCTACHLOROCYCLOPENTENE [# 021 Code: C58 Rev: 07/01/96 R# 186]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 706-78-5

HEXACHLOROBENZENE [# 018 Code: C66 Rev: 07/01/96 R# 187]
PQL: 0.0001 mg/L Min DL: 0.0001 mg/L RMCL: 0.001 mg/L REGULATORY MCL CAS: 118-74-1

CARBOXIN [# 041 Code: CARBOXIN Rev: 07/01/96 R# 188]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 5234-68-4

CHLOROTHALONIL [# 033 Code: CHLRTHNL Rev: 07/01/96 R# 189]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 1897-45-6

CHLORDANE,alpha [# 008 Code: CLRDNA Rev: 07/01/96 R# 190]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.002 mg/L REGULATORY MCL CAS: 5103-71-9

CHLORDANE,gamma- [# 009 Code: CLRDNG Rev: 07/01/96 R# 191]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.002 mg/L REGULATORY MCL CAS: 5103-74-2

CYCLOATE [# 039 Code: CYCLOATE Rev: 07/01/96 R# 192]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 1134-23-2

CYPRAZINE [# 054 Code: CYPRAZIN Rev: 07/01/96 R# 193]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 22936-86-3

TEST CODE | TEST FEE | METHOD INFORMATION - SOC | LAB GROUP

CXPT (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

DDD,4,4`- [# 010 Code: DDD44 Rev: 07/01/96 R# 194]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 72-54-8

DDE,4,4`- [# 011 Code: DDE44 Rev: 07/01/96 R# 195]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 72-55-9

DDT,4,4`- [# 012 Code: DDT44 Rev: 07/01/96 R# 196]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 50-29-3

DIPHENAMID [# 042 Code: DIPHNMID Rev: 07/01/96 R# 197]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 957-51-7

DIELDRIN [# 030 Code: DLDRN Rev: 07/01/96 R# 198]
PQL: 0.0005 mg/L Min DL: 0.0005 mg/L RMCL: NONE ESTABLISHED CAS: 60-57-1

ENDOSULFAN,alpha [# 013 Code: ENDOSLA Rev: 07/01/96 R# 199]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 959-98-8

ENDOSULFAN,beta [# 014 Code: ENDOSLB Rev: 07/01/96 R# 200]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 33213-65-9

ENDRIN ALDEHYDE [# 015 Code: ENDRHYD Rev: 07/01/96 R# 201]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 7421-93-4

ENDRIN [# 001 Code: ENDRIN Rev: 07/01/96 R# 202]
PQL: 0.00005 mg/L Min DL: 0.00005 mg/L RMCL: 0.002 mg/L REGULATORY MCL CAS: 72-20-8

EPTC [# 055 Code: EPTAM Rev: 07/01/96 R# 203]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 759-94-4

HEXAZINONE [# 043 Code: HEXAZNON Rev: 07/01/96 R# 204]
PQL: 0.003 mg/L Min DL: 0.003 mg/L RMCL: NONE ESTABLISHED CAS: 51235-04-2

HEPTACHLOR [# 016 Code: HPTCL Rev: 07/01/96 R# 205]
PQL: 0.00008 mg/L Min DL: 0.00008 mg/L RMCL: 0.0004 mg/L REGULATORY MCL
CAS: 76-44-8

HEPTACHLOR EPOXIDE [# 017 Code: HPTCLOX Rev: 07/01/96 R# 206]
PQL: 0.00004 mg/L Min DL: 0.00004 mg/L RMCL: 0.0002 mg/L REGULATORY MCL
CAS: 1024-57-3

METOLACHLOR [# 044 Code: METLCHLR Rev: 07/01/96 R# 207]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 51218-45-2

METHOXYCHLOR [# 003 Code: METOXCL Rev: 07/01/96 R# 208]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.04 mg/L REGULATORY MCL CAS: 72-43-5

TEST CODE | TEST FEE | METHOD INFORMATION - SOC | LAB GROUP

CXPT (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

METRIBUZIN [# 045 Code: METRBUZN Rev: 07/01/96 R# 209]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 21087-64-9

MOLINATE [# 058 Code: MOLINATE Rev: 07/01/96 R# 210]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 2212-67-1

POLYBROMINATED BIPHENYLS [# 022 Code: PBB Rev: 07/01/96 R# 211]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 59536-65-1

PCB (AROCLOR 1016) [# 023 Code: PCB1016 Rev: 07/01/96 R# 212]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.0005 mg/L REGULATORY MCL
CAS: 12674-11-2

PCB (AROCLOR 1221) [# 024 Code: PCB1221 Rev: 07/01/96 R# 213]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.0005 mg/L REGULATORY MCL
CAS: 11104-28-2

PCB (AROCLOR 1232) [# 025 Code: PCB1232 Rev: 07/01/96 R# 214]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.0005 mg/L REGULATORY MCL
CAS: 11141-16-5

PCB (AROCLOR 1242) [# 026 Code: PCB1242 Rev: 07/01/96 R# 215]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.0005 mg/L REGULATORY MCL
CAS: 53469-21-9

PCB (AROCLOR 1248) [# 027 Code: PCB1248 Rev: 07/01/96 R# 216]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.0005 mg/L REGULATORY MCL
CAS: 12672-29-6

PCB (AROCLOR 1254) [# 028 Code: PCB1254 Rev: 07/01/96 R# 217]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.0005 mg/L REGULATORY MCL
CAS: 11097-69-1

PCB (AROCLOR 1260) [# 029 Code: PCB1260 Rev: 07/01/96 R# 218]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.0005 mg/L REGULATORY MCL
CAS: 11096-82-5

PROMETON [# 046 Code: PROMETON Rev: 07/01/96 R# 219]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 1610-18-0

PRONAMIDE [# 047 Code: PRONAMID Rev: 07/01/96 R# 220]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 23950-58-5

PROPACHLOR [# 052 Code: PROPACHL Rev: 07/01/96 R# 221]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 1918-16-7

PROPAZINE [# 048 Code: PROPAZIN Rev: 07/01/96 R# 222]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 139-40-2

SIMAZINE [# 049 Code: SIMAZINE Rev: 07/01/96 R# 223]

PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.004 mg/L REGULATORY MCL CAS: 122-34-9

TEST CODE | TEST FEE | METHOD INFORMATION - SOC | LAB GROUP

CXPT (CONTINUED)

REPORTED ANALYTES FOR TEST PROCEDURE:

TEBUTHIURON [# 050 Code: TEBTHURN Rev: 07/01/96 R# 224]
PQL: 0.005 mg/L Min DL: 0.005 mg/L RMCL: NONE ESTABLISHED CAS: 34014-18-1

TERBACIL [# 035 Code: TERBACIL Rev: 07/01/96 R# 225]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 5902-51-2

TOXAPHENE [# 031 Code: TOXAPHN Rev: 07/01/96 R# 226]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: 0.003 mg/L REGULATORY MCL CAS: 8001-35-2

TRIFLURALIN [# 056 Code: TRIFLAN Rev: 07/01/96 R# 227]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 1582-09-8

TEST CODE	TEST FEE	METHOD INFORMATION - BACTI LAB GROUP

BIRON 0.00 IRON BACTERIA BY BART TEST [Ref.: IN-HOUSE]
Sample Kit: 32 Holding Limit: NONE Lab Group: BACTI [Rec# 1 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

IRON BACTERIA ANALYSIS [# 001 Code: BIRON Rev: 07/01/96 R# 1]
PQL: Min DL: RMCL: NONE ESTABLISHED CAS: BIRON-00-B

BPTC 12.00 DRINKING WATER COLIFORMS [Ref.: SM9223B]
Sample Kit: 30 Holding Limit: 1 days Lab Group: BACTI [Rec# 3 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

COLIFORM ORGANISMS PER 100ml [# 001 Code: PTC Rev: 07/01/96 R# 3]
PQL: Min DL: RMCL: NONE ESTABLISHED CAS: TC-00-B

BSPC 12.00 HETEROTROPHIC PLATE COUNT [Ref.: SIMPLATE]
Sample Kit: 30 Holding Limit: 1 days Lab Group: BACTI [Rec# 4 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

HETEROTROPHIC PLATE COUNT PER 1ml [# 001 Code: 24 HR Rev: 07/01/96 R# 4]
PQL: MPN Min DL: MPN RMCL: NONE ESTABLISHED CAS:

NPEC 15.00 E COLI FROM SURFACE/WASTEWATER [Ref.: EPA1103.1]
Sample Kit: 30 Holding Limit: 1 days Lab Group: BACTI [Rec# 84 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

E. COLI PER 100ml [# 001 Code: EC COUNT Rev: 07/01/96 R# 298]
PQL: Min DL: RMCL: NONE ESTABLISHED CAS: EC-00-B

NPFC 15.00 FECAL COLIFORMS FOR SURFACE/WASTEWATER [Ref.: SM9222D]
Sample Kit: 30 Holding Limit: 1 days Lab Group: BACTI [Rec# 85 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

FECAL COLIFORM ORGANISMS PER 100ml [# 001 Code: FC COUNT Rev: 07/01/96 R# 299]
PQL: Min DL: RMCL: NONE ESTABLISHED CAS: FC-00-B

NPFS 5.00 FECAL STREPTOCOCCI FOR SURFACE/WASTEWATER [Ref.: SM9230C]
Sample Kit: 30 Holding Limit: 1 days Lab Group: BACTI [Rec# 86 Rev. 07/01/96]

REPORTED ANALYTES FOR TEST PROCEDURE:

TEST CODE	TEST FEE	METHOD INFORMATION - BACTI LAB GROUP
NPPC	12.00	SWIMMING POOL HETEROTROPHIC PLATE COUNT [Ref.: SIMPLATE]
		Sample Kit: 30 Holding Limit: 1 days Lab Group: BACTI [Rec# 87 Rev. 07/01/96]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		HETEROTROPHIC PLATE COUNT PER 1ml [# 001 Code: SPC Rev: 07/01/96 R# 300]
		PQL: MPN Min DL: MPN RMCL: NONE ESTABLISHED CAS:
NSEC	15.00	E. COLI FROM SEDIMENTS [Ref.: EPA1103.1]
		Sample Kit: 30 Holding Limit: 3 days Lab Group: BACTI [Rec# 88 Rev. 07/01/96]
		REPORTED ANALYTES FOR TEST PROCEDURE:
NSFC	15.00	FECAL COLIFORMS FOR SEDIMENTS [Ref.: SM9222D]
		Sample Kit: 30 Holding Limit: 3 days Lab Group: BACTI [Rec# 89 Rev. 07/01/96]
		REPORTED ANALYTES FOR TEST PROCEDURE:
NSFS	15.00	FECAL STREPTOCOCCI IN SEDIMENTS [Ref.: SM9230C]
		Sample Kit: 30 Holding Limit: 3 days Lab Group: BACTI [Rec# 90 Rev. 07/01/96]
		REPORTED ANALYTES FOR TEST PROCEDURE:
NSPC	12.00	SWIMMING POOLS WATER COLIFORM [Ref.: SM9221D9222B]
		Sample Kit: 30 Holding Limit: 1 days Lab Group: BACTI [Rec# 91 Rev. 07/01/96]
		REPORTED ANALYTES FOR TEST PROCEDURE:
		COLIFORM ORGANISMS PER 100ml [# 001 Code: PTC Rev: 07/01/96 R# 301]
		PQL: Min DL: RMCL: NONE ESTABLISHED CAS: TC-00-B
SID	0.00	SOLIDS IDENTIFICATION INVESTIGATION [Ref.: IN-HOUSE]
		Sample Kit: 32 Holding Limit: NONE Lab Group: BACTI [Rec# 92 Rev. 07/01/96]
		REPORTED ANALYTES FOR TEST PROCEDURE:

TEST CODE	TEST FEE	METHOD INFORMATION - GROUP LAB GROUP
B	12.00	BPTC

REPORTED ANALYTES FOR TEST PROCEDURE:

COLIFORM ORGANISMS PER 100ml [# 001 Code: PTC Rev: 07/01/96 R# 3]
PQL: Min DL: RMCL: NONE ESTABLISHED CAS: TC-00-B

TEST CODE	TEST FEE	METHOD INFORMATION - GROUP LAB GROUP
CCUB	21.00	CPB CMCU

REPORTED ANALYTES FOR TEST PROCEDURE:

LEAD BY ICP/MS [# 001 Code: PB Rev: 12/10/99 R# 38]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 7439-92-1

COPPER [# 001 Code: CU Rev: 01/04/00 R# 29]
PQL: 0.05 mg/L Min DL: 0.05 mg/L RMCL: NONE ESTABLISHED CAS: 7440-50-8

TEST CODE	TEST FEE	METHOD INFORMATION - GROUP LAB GROUP
CDHB	190.00	CXHB CDCPA

REPORTED ANALYTES FOR TEST PROCEDURE:

2,4,5-T [# 003 Code: 245T Rev: 07/01/96 R# 106]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 93-76-5

2,4,5-TP (SILVEX) [# 005 Code: 245TP Rev: 07/01/96 R# 107]
PQL: 0.0003 mg/L Min DL: 0.0003 mg/L RMCL: 0.05 mg/L REGULATORY MCL CAS: 93-72-1

2,4-D [# 008 Code: 24D Rev: 07/01/96 R# 108]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: 0.07 mg/L REGULATORY MCL CAS: 94-75-7

ACIFLUORFEN [# 006 Code: ACFLO Rev: 07/01/96 R# 109]
PQL: 0.004 mg/L Min DL: 0.004 mg/L RMCL: NONE ESTABLISHED CAS: 50594-66-6

BENTAZON [# 002 Code: BENTAZON Rev: 07/01/96 R# 110]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 25057-89-0

DCPA, MONO [# 010 Code: DACTHAL Rev: 07/01/96 R# 111]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 867-54-7

DICAMBA [# 007 Code: DICAMBA Rev: 07/01/96 R# 112]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: NONE ESTABLISHED CAS: 1918-00-9

DINOSEB [# 004 Code: DINOSEB Rev: 07/01/96 R# 113]
PQL: 0.0003 mg/L Min DL: 0.0003 mg/L RMCL: 0.007 mg/L REGULATORY MCL CAS: 88-85-7

PENTACHLOROPHENOL [# 009 Code: PCP Rev: 07/01/96 R# 114]
PQL: 0.00006 mg/L Min DL: 0.00006 mg/L RMCL: 0.001 mg/L REGULATORY MCL CAS: 87-86-5

PICLORAM [# 001 Code: PICLORAM Rev: 07/01/96 R# 115]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: 0.5 mg/L REGULATORY MCL CAS: 1918-02-1

TEST CODE	TEST FEE	METHOD INFORMATION - GROUP LAB GROUP
CDHB	(CONTINUED)	

REPORTED ANALYTES FOR TEST PROCEDURE:

TOTAL DCPA, MONO & DI-ACID DEGRADATES [# 001 Code: DCPA Rev: 06/05/01 R# 22]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 1861-32-1

CLMT	64.00	CBE CNI CTL CSB

REPORTED ANALYTES FOR TEST PROCEDURE:

BERYLLIUM [# 001 Code: BE Rev: 07/01/96 R# 8]
PQL: 0.0004 mg/L Min DL: 0.0004 mg/L RMCL: 0.004 mg/L REGULATORY MCL CAS: 7440-41-7

NICKEL [# 001 Code: NI Rev: 07/01/96 R# 35]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: 0.1 mg/L REGULATORY MCL CAS: 7440-02-0

THALLIUM [# 001 Code: TL Rev: 07/01/96 R# 71]
PQL: 0.0002 mg/L Min DL: 0.0002 mg/L RMCL: 0.002 mg/L REGULATORY MCL CAS: 7440-28-0

ANTIMONY [# 001 Code: SB Rev: 07/01/96 R# 63]
PQL: 0.0006 mg/L Min DL: 0.0006 mg/L RMCL: 0.006 mg/L REGULATORY MCL CAS: 7440-36-0

CMET	132.00	CBA CAS CCR CCD CPB CSE CCU CZN CMN CFE CHG

REPORTED ANALYTES FOR TEST PROCEDURE:

BARIUM [# 001 Code: BA Rev: 07/01/96 R# 7]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: 2 mg/L REGULATORY MCL CAS: 7440-39-3

ARSENIC [# 002 Code: AS Rev: 04/15/97 R# 6]
PQL: 0.002 mg/L Min DL: 0.002 mg/L RMCL: 0.01 mg/L REGULATORY MCL CAS: 7440-38-2

CHROMIUM [# 001 Code: CR Rev: 07/01/96 R# 15]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: 0.1 mg/L REGULATORY MCL CAS: 7440-47-3

CADMIUM [# 001 Code: CD Rev: 07/01/96 R# 10]
PQL: 0.0003 mg/L Min DL: 0.0003 mg/L RMCL: 0.005 mg/L REGULATORY MCL CAS: 7440-43-9

LEAD BY ICP/MS [# 001 Code: PB Rev: 12/10/99 R# 38]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: NONE ESTABLISHED CAS: 7439-92-1

SELENIUM [# 001 Code: SE Rev: 07/01/96 R# 64]
PQL: 0.001 mg/L Min DL: 0.001 mg/L RMCL: 0.05 mg/L REGULATORY MCL CAS: 7782-49-2

COPPER [# 001 Code: CU Rev: 11/01/01 R# 16]
PQL: 0.05 mg/L Min DL: 0.05 mg/L RMCL: NONE ESTABLISHED CAS: 7440-50-8

TEST CODE	TEST FEE	METHOD INFORMATION - GROUP LAB GROUP
CMET	(CONTINUED)	

REPORTED ANALYTES FOR TEST PROCEDURE:

ZINC		[# 001 Code: ZN	Rev: 11/01/01 R# 296]
PQL: 0.01 mg/L	Min DL: 0.01 mg/L	RMCL: NONE ESTABLISHED	CAS: 7440-66-6
MANGANESE		[# 001 Code: MN	Rev: 11/01/01 R# 31]
PQL: 0.01 mg/L	Min DL: 0.01 mg/L	RMCL: NONE ESTABLISHED	CAS: 7439-96-5
IRON		[# 001 Code: FE	Rev: 11/01/01 R# 24]
PQL: 0.01 mg/L	Min DL: 0.01 mg/L	RMCL: NONE ESTABLISHED	CAS: 7439-89-6
MERCURY		[# 002 Code: HG	Rev: 04/16/97 R# 26]
PQL: 0.0001 mg/L	Min DL: 0.0001 mg/L	RMCL: 0.002 mg/L REGULATORY MCL	CAS: 7439-97-6

CMET2	176.00	CHG CBA CSE CAS CCR CCD CPB CBE CTL CNI CSB

REPORTED ANALYTES FOR TEST PROCEDURE:

MERCURY		[# 002 Code: HG	Rev: 04/16/97 R# 26]
PQL: 0.0001 mg/L	Min DL: 0.0001 mg/L	RMCL: 0.002 mg/L REGULATORY MCL	CAS: 7439-97-6
BARIUM		[# 001 Code: BA	Rev: 07/01/96 R# 7]
PQL: 0.01 mg/L	Min DL: 0.01 mg/L	RMCL: 2 mg/L REGULATORY MCL	CAS: 7440-39-3
SELENIUM		[# 001 Code: SE	Rev: 07/01/96 R# 64]
PQL: 0.001 mg/L	Min DL: 0.001 mg/L	RMCL: 0.05 mg/L REGULATORY MCL	CAS: 7782-49-2
ARSENIC		[# 002 Code: AS	Rev: 04/15/97 R# 6]
PQL: 0.002 mg/L	Min DL: 0.002 mg/L	RMCL: 0.01 mg/L REGULATORY MCL	CAS: 7440-38-2
CHROMIUM		[# 001 Code: CR	Rev: 07/01/96 R# 15]
PQL: 0.01 mg/L	Min DL: 0.01 mg/L	RMCL: 0.1 mg/L REGULATORY MCL	CAS: 7440-47-3
CADMIUM		[# 001 Code: CD	Rev: 07/01/96 R# 10]
PQL: 0.0003 mg/L	Min DL: 0.0003 mg/L	RMCL: 0.005 mg/L REGULATORY MCL	CAS: 7440-43-9
LEAD BY ICP/MS		[# 001 Code: PB	Rev: 12/10/99 R# 38]
PQL: 0.001 mg/L	Min DL: 0.001 mg/L	RMCL: NONE ESTABLISHED	CAS: 7439-92-1
BERYLLIUM		[# 001 Code: BE	Rev: 07/01/96 R# 8]
PQL: 0.0004 mg/L	Min DL: 0.0004 mg/L	RMCL: 0.004 mg/L REGULATORY MCL	CAS: 7440-41-7
THALLIUM		[# 001 Code: TL	Rev: 07/01/96 R# 71]
PQL: 0.0002 mg/L	Min DL: 0.0002 mg/L	RMCL: 0.002 mg/L REGULATORY MCL	CAS: 7440-28-0

TEST CODE	TEST FEE	METHOD INFORMATION - GROUP LAB GROUP
CMET2	(CONTINUED)	

REPORTED ANALYTES FOR TEST PROCEDURE:

NICKEL [# 001 Code: NI Rev: 07/01/96 R# 35]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: 0.1 mg/L REGULATORY MCL CAS: 7440-02-0

ANTIMONY [# 001 Code: SB Rev: 07/01/96 R# 63]
PQL: 0.0006 mg/L Min DL: 0.0006 mg/L RMCL: 0.006 mg/L REGULATORY MCL CAS: 7440-36-0

CMIN	110.00	CTALK CRM CSI CK CCON CPH CCA CMG CNA

REPORTED ANALYTES FOR TEST PROCEDURE:

TOTAL ALKALINITY AS CaCO3 [# 001 Code: TOTAL AL Rev: 05/15/97 R# 69]
PQL: 1 mg/L Min DL: 1 mg/L RMCL: NONE ESTABLISHED CAS: TALK-00-C

FLUORIDE (AUTOMATED) [# 004 Code: AF Rev: 05/05/97 R# 57]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: 4.0 mg/L REGULATORY MCL CAS: 16984-48-8

HARDNESS AS CaCO3 [# 005 Code: AHA Rev: 05/05/97 R# 58]
PQL: 10 mg/L Min DL: 10 mg/L RMCL: NONE ESTABLISHED CAS: HARD-00-C

CHLORIDE (AUTOMATED) [# 003 Code: CL Rev: 05/05/97 R# 59]
PQL: 4 mg/L Min DL: 4 mg/L RMCL: NONE ESTABLISHED CAS: 7647-14-5

NITRITE AS N (AUTOMATED) [# 002 Code: NO2 Rev: 05/05/97 R# 60]
PQL: 0.05 mg/L Min DL: 0.05 mg/L RMCL: 1 mg/L REGULATORY MCL CAS: 14797-65-0

NITRATE AS N (AUTOMATED) [# 001 Code: NO3 Rev: 05/05/97 R# 61]
PQL: 0.4 mg/L Min DL: 0.4 mg/L RMCL: 10 mg/L REGULATORY MCL CAS: 14797-55-8

SULFATE (AUTOMATED) [# 006 Code: SO4 Rev: 05/05/97 R# 62]
PQL: 5 mg/L Min DL: 5 mg/L RMCL: NONE ESTABLISHED CAS: 14808-79-8

SILICA AS SiO2 [# 001 Code: SI Rev: 07/01/96 R# 65]
PQL: 0.2 mg/L Min DL: 0.2 mg/L RMCL: NONE ESTABLISHED CAS: 7440-21-3

POTASSIUM [# 001 Code: K Rev: 07/01/96 R# 28]
PQL: 0.03 mg/L Min DL: 0.03 mg/L RMCL: NONE ESTABLISHED CAS: 7440-09-7

CONDUCTIVITY [# 001 Code: CON Rev: 07/01/96 R# 14]
PQL: 1 umhos Min DL: 1 umhos RMCL: NONE ESTABLISHED CAS: COND-00-C

pH [# 001 Code: PH Rev: 07/01/96 R# 39]
PQL: 2.0 Min DL: 2.0 RMCL: NONE ESTABLISHED CAS: PH-00-C

TEST CODE	TEST FEE	METHOD INFORMATION - GROUP LAB GROUP
CMIN	(CONTINUED)	

REPORTED ANALYTES FOR TEST PROCEDURE:

CALCIUM		[# 002 Code: CA	Rev: 04/15/97 R# 9]
PQL: 0.3 mg/L	Min DL: 0.3 mg/L	RMCL: NONE ESTABLISHED	CAS: 7440-70-2
MAGNESIUM		[# 002 Code: MG	Rev: 04/16/97 R# 30]
PQL: 0.1 mg/L	Min DL: 0.1 mg/L	RMCL: NONE ESTABLISHED	CAS: 7439-95-4
SODIUM		[# 002 Code: NA	Rev: 04/16/97 R# 33]
PQL: 0.1 mg/L	Min DL: 0.1 mg/L	RMCL: NONE ESTABLISHED	CAS: 7440-23-5

CORR	73.00	CTALK CCON CPO4 CCA CMG CNA

REPORTED ANALYTES FOR TEST PROCEDURE:

TOTAL ALKALINITY AS CaCO3		[# 001 Code: TOTAL AL	Rev: 05/15/97 R# 69]
PQL: 1 mg/L	Min DL: 1 mg/L	RMCL: NONE ESTABLISHED	CAS: TALK-00-C
CONDUCTIVITY		[# 001 Code: CON	Rev: 07/01/96 R# 14]
PQL: 1 umhos	Min DL: 1 umhos	RMCL: NONE ESTABLISHED	CAS: COND-00-C
ORTHO-PHOSPHATE AS P		[# 001 Code: PO4	Rev: 07/01/96 R# 47]
PQL: 0.1 mg/L	Min DL: 0.1 mg/L	RMCL: NONE ESTABLISHED	CAS: 7664-38-2
CALCIUM		[# 002 Code: CA	Rev: 04/15/97 R# 9]
PQL: 0.3 mg/L	Min DL: 0.3 mg/L	RMCL: NONE ESTABLISHED	CAS: 7440-70-2
MAGNESIUM		[# 002 Code: MG	Rev: 04/16/97 R# 30]
PQL: 0.1 mg/L	Min DL: 0.1 mg/L	RMCL: NONE ESTABLISHED	CAS: 7439-95-4
SODIUM		[# 002 Code: NA	Rev: 04/16/97 R# 33]
PQL: 0.1 mg/L	Min DL: 0.1 mg/L	RMCL: NONE ESTABLISHED	CAS: 7440-23-5

CPM1	20.00	CCU CZN CMN CFE

REPORTED ANALYTES FOR TEST PROCEDURE:

COPPER		[# 001 Code: CU	Rev: 11/01/01 R# 16]
PQL: 0.05 mg/L	Min DL: 0.05 mg/L	RMCL: NONE ESTABLISHED	CAS: 7440-50-8
ZINC		[# 001 Code: ZN	Rev: 11/01/01 R# 296]
PQL: 0.01 mg/L	Min DL: 0.01 mg/L	RMCL: NONE ESTABLISHED	CAS: 7440-66-6
MANGANESE		[# 001 Code: MN	Rev: 11/01/01 R# 31]
PQL: 0.01 mg/L	Min DL: 0.01 mg/L	RMCL: NONE ESTABLISHED	CAS: 7439-96-5

TEST CODE	TEST FEE	METHOD INFORMATION - GROUP LAB GROUP
CPM1	(CONTINUED)	

REPORTED ANALYTES FOR TEST PROCEDURE:

IRON [# 001 Code: FE Rev: 11/01/01 R# 24]
PQL: 0.01 mg/L Min DL: 0.01 mg/L RMCL: NONE ESTABLISHED CAS: 7439-89-6

CPM2	28.00	CCA CMG CNA

REPORTED ANALYTES FOR TEST PROCEDURE:

CALCIUM [# 002 Code: CA Rev: 04/15/97 R# 9]
PQL: 0.3 mg/L Min DL: 0.3 mg/L RMCL: NONE ESTABLISHED CAS: 7440-70-2

MAGNESIUM [# 002 Code: MG Rev: 04/16/97 R# 30]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: NONE ESTABLISHED CAS: 7439-95-4

SODIUM [# 002 Code: NA Rev: 04/16/97 R# 33]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: NONE ESTABLISHED CAS: 7440-23-5

N	15.00	NPFC

REPORTED ANALYTES FOR TEST PROCEDURE:

FECAL COLIFORM ORGANISMS PER 100ml [# 001 Code: FC COUNT Rev: 07/01/96 R# 299]
PQL: Min DL: RMCL: NONE ESTABLISHED CAS: FC-00-B

NSED	45.00	NSEC NSFC NSFS

REPORTED ANALYTES FOR TEST PROCEDURE:

R	14.00	CRA

REPORTED ANALYTES FOR TEST PROCEDURE:

FLUORIDE (AUTOMATED) [# 005 Code: AF Rev: 07/01/96 R# 48]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: 4.0 mg/L REGULATORY MCL CAS: 16984-48-8

IRON (AUTOMATED) [# 007 Code: AFE Rev: 07/01/96 R# 49]
PQL: 0.1 mg/L Min DL: 0.1 mg/L RMCL: NONE ESTABLISHED CAS: 7439-89-6

HARDNESS AS CaCO3 [# 006 Code: AHA Rev: 07/01/96 R# 50]
PQL: 10 mg/L Min DL: 10 mg/L RMCL: NONE ESTABLISHED CAS: HARD-00-C

SODIUM (AUTOMATED) [# 009 Code: ANA Rev: 07/01/96 R# 51]
PQL: 5 mg/L Min DL: 5 mg/L RMCL: NONE ESTABLISHED CAS: 7440-23-5

TEST CODE	TEST FEE	METHOD INFORMATION - GROUP	LAB GROUP
R	(CONTINUED)		

REPORTED ANALYTES FOR TEST PROCEDURE:

CHLORIDE (AUTOMATED)		[# 004 Code: CL	Rev: 07/01/96 R# 52]
PQL: 4 mg/L	Min DL: 4 mg/L	RMCL: NONE ESTABLISHED	CAS: 7647-14-5
NITRITE AS N (AUTOMATED)		[# 003 Code: NO2	Rev: 07/01/96 R# 53]
PQL: 0.05 mg/L	Min DL: 0.05 mg/L	RMCL: 1 mg/L REGULATORY MCL	CAS: 14797-65-0
NITRATE AS N (AUTOMATED)		[# 001 Code: NO3	Rev: 07/01/96 R# 54]
PQL: 0.4 mg/L	Min DL: 0.4 mg/L	RMCL: 10 mg/L REGULATORY MCL	CAS: 14797-55-8
SULFATE (AUTOMATED)		[# 008 Code: SO4	Rev: 07/01/96 R# 55]
PQL: 5 mg/L	Min DL: 5 mg/L	RMCL: NONE ESTABLISHED	CAS: 14808-79-8

SW	12.00	NSPC

REPORTED ANALYTES FOR TEST PROCEDURE:

COLIFORM ORGANISMS PER 100ml		[# 001 Code: PTC	Rev: 07/01/96 R# 301]
PQL:	Min DL:	RMCL: NONE ESTABLISHED	CAS: TC-00-B

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

LABORATORY SERVICES SECTION
ENVIRONMENTAL LABORATORY
QUALITY ASSURANCE PROGRAM PLAN

APPENDIX F

Drinking Water Sampling Procedures Manual

QAPP-Ver2.0, Revison 0
May 5, 2003

Drinking Water Unit
Sampling Procedures Manual

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Introduction

It is the sample collector's responsibility to use the correct sample container and sampling technique. If the wrong unit or technique are used, the sample's validity is questionable. These sampling errors may result in monitoring violations, a waste of the laboratory's time, and the squandering of the state's resources.

Although many of the contaminants utilize the same sampling unit, similar contaminants, such as the total trihalomethanes (TTHM) and the volatile organic chemicals (VOC), may use different sampling units. The difference between similar units is the preservatives in the container prior to sample collection or added to the sample container during sample collection. Typically, the preservatives are dechlorinating agents. These chemicals must remain in the bottle and must not be rinsed out. If the chemicals spill on to your skin or clothing, the effected area must be immediately flushed with water for several minutes.

In the attached "MDEQ Analysis Section Testing Catalog", the proper sample container is listed for each contaminant. After you receive your bottle(s), check to see that the sample unit label on the unit box or container shows the same unit number as indicated in the catalog. Different tests may require different sample units.

Following the proper sampling protocol is just as important as selecting the right container. The requirements of the lead and copper rules are an excellent example where the technique requires a first draw sample versus the standard of letting the water run for several minutes prior to sample collection. Other examples include the choice of a chlorinated or unchlorinated sampling tap. For a few of the techniques, such as for the TTHM and VOC, the sample collector may be required to obtain a trip or field blank from the laboratory prior to leaving on his/her sampling trip. Also, the collector must be aware of maximum transit times permitted between sample collection and its delivery to the laboratory and requirements to cool the sample, as described in the catalog. Coolers are available for the use of the DEQ staff for the purpose of transporting the samples from the sampling site to the laboratory.

The issues on sample collection are discussed in other sections of this manual. Tables 1 and 2 have been included in this manual to allow a comparison between the procedures used by the MDEQ laboratory and the approved U.S. EPA techniques. Also, these tables should be consulted for the selection of the proper sampling unit. Either Table 1 or 2 and the "MDEQ Water Analysis Section Testing Catalog" should be consulted for the correct bottles to be used and the maximum holding times allowed by the laboratory.

Training

At the bare minimum, the sample collector must read and understand the directions for each type of sample. The collector may need to be familiar with field chlorine residual monitoring, aseptic sampling procedures, safe procedures for handling corrosive materials, and specific on-site training regarding sample tap locations.

Training on sample collection is available through many of the MDEQ/Michigan Section AWWA short courses, which include the Michigan State University Bacteriology Short Course, the Limited Treatment Short Course, Distribution System Operator Short

Course, and the Complete Treatment Short Courses I and II. The matter has been discussed in a few of the recent Michigan Section AWWA Regional Meetings and in several of the MDEQ training manuals. Also, sample collection training is directly available from the senior MDEQ staff members.

Sample Unit Directions

Directions for Sample Unit 30

1. Do not open the bottle until ready to collect the sample. Do not touch the inside of the cap or bottle. Retain the cap in your hand, and do not set the cap down.

2. Do not rinse the bottle with sample. The bottle contains sodium thiosulfate for the deactivation of chlorine. If not collecting sample from a tap (lake, pool, etc.), plunge bottle mouth down and move in continuous arc down and back up from water. Discard top half-inch of sample. Go to step three (3) if using a sample tap.

3. Select a clean faucet and remove such attachments as aerators, dishwasher connectors, etc. Allow water to run for ten minutes (until cold) at full flow from the sampling tap. Reduce flow to avoid splashing, and collect the sample directly into the bottle. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the bottle. Fill bottle only to the bottom of the neck. Replace cap.

Directions for Sample Units 32, 33 and 36ME

1. Do not open the bottle until ready to collect the sample. Do not touch the inside of the cap or bottle.

2. Do not rinse the bottle with sample. Select a clean faucet and remove such attachments as aerators, dishwasher connectors, etc. Allow water to run for ten minutes (until cold) at full flow at the sampling point.

3. Reduce flow and collect the sample directly into the bottle. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the bottle. Avoid splashing. Fill bottle to the bottom of the neck. Replace cap.

Directions for Sample Unit 36CC

1. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle.

2. Sampling point should not have been used for a minimum of six hours prior to sampling. Do not flush the sample tap before sample collection.

3. Place bottle mouth below tap and turn on water filling bottle to the bottom of the neck. Replace cap.

Directions for Sample Unit 36AC

1. This testing unit contains a diluted sulfuric acid preservative in the sample bottle and caution should be exercised. Tap sample bottle in upright position to drain preservatives from cap. Do not rinse bottle before collection. If preservative is spilled, immediately flush skin with large amounts of water.

2. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle.

3. Select a clean faucet avoiding such attachments as aerators, dishwasher connectors, etc. Allow water to run for ten minutes (until cold) at full flow from the sampling tap.

4. Reduce flow to avoid splashing, and collect the sample directly into the bottle. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the bottle. Fill bottle to one-half inch below the bottom of the neck. Cap and invert five times to mix sample with the preservative.

Directions for Sample Unit 36CN

1. Use a field chlorine residual test kit to confirm the absence of a free or combined chlorine residual. If no chlorine residual is present, proceed with the collection of the sample using Unit 36CN. If a chlorine residual is present, Unit 36CNa should be used.
2. This testing unit contains caustic 10N sodium hydroxide preservatives and caution should be exercised. Tap unit in upright position to drain preservatives from cap. Do not rinse bottle before collection. If preservative is spilled, immediately flush skin with large amounts of water.

3. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle.

4. Select a clean faucet avoiding such attachments as aerators, dishwasher connectors, etc. Allow water to run for ten minutes (or until cold) at full flow at the sampling point.

5. Reduce flow to avoid splashing, and collect the sample directly into the bottle. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the bottle. Fill bottle to the bottom of the neck. Cap and invert five times to mix sample with preservative.

Directions for Sample Unit 36CNa

1. This testing unit contains ascorbic acid. A separate vial with caustic 10N sodium hydroxide preservative is needed with this sampling unit. Caution should be exercised in the collection of the sample. Tap unit in upright position to drain any of the acid preservative from cap. Do not rinse bottle before collection. If the acid preservative is spilled, immediately flush skin with large amounts of water.

2. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle.

3. Select a clean faucet avoiding such attachments as aerators, dishwasher connectors, etc. Allow water to run for ten minutes at full flow at the sampling point.

4. Reduce flow to avoid splashing, and collect the sample directly into the bottle. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the bottle. Fill bottle to one-half inch below the bottom of the neck. Cap and invert five times to mix sample with preservatives.

5. Remove sample bottle cap and add all sodium hydroxide preservative solution in vial to sample bottle. Recap and invert five times to mix. Immediately flush skin with large amounts of water if the preservative is spilled.

Directions for Sampling Units 36LP

1. Use a field chlorine residual test kit to confirm the absence of a free or a total chlorine residual. If no chlorine residual is present, proceed with the collection of the sample using Unit 36LP. If a chlorine residual is present, collect the sample using unit 36LPa.

2. This testing unit contains monochloracetic acid as a preservative and caution should be exercised. Shake unit in upright position to drain preservatives from cap. Do not rinse bottle before collection. Immediately flush skin with large amounts of water if spill occurs.

3. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle.

4. Do not rinse the bottle with sample. Select a clean faucet avoiding such attachments as aerators, dishwasher connectors, etc. Allow water to run for ten minutes at full flow at the sampling point.

5. Reduce flow and collect the sample directly into the bottle. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the bottle. Avoid splashing. Fill bottle to the bottom of the neck. Cap and invert five times to mix sample with preservatives.

Directions for Sampling Unit 36LPa

1. Use a field chlorine residual test kit to confirm the absence of a free or a total chlorine residual. If a chlorine residual is present, proceed with the collection of the sample using Unit 36LPa. If a chlorine residual is absent, use Unit 36LP to collect the sample.

2. The test unit contains sodium thiosulfate as a preservative and caution should be exercised. A separate vial containing monochloracetic acid is required with this unit. Shake unit in upright position to drain preservatives from cap of the sample bottle. Do not rinse bottle before collection. Immediately flush skin with large amounts of water if spill occurs.

3. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle.

4. Do not rinse the bottle with sample. Select a clean faucet avoiding such attachments as aerators, dishwasher connectors, etc. Allow water to run for ten minutes at full flow at the sampling point.
5. Reduce flow and collect the sample directly into the bottle. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the bottle. Avoid splashing. Fill bottle to the bottom of the neck. Cap and invert five times to mix sample with preservative.

6. Remove the bottle's cap and add the contents of the accompanying vial, which contains monochloracetic acid. Recap and invert five times to mix the sample with the preservatives. In the event of a spill, immediately flush the skin with large amounts of water.

Directions for Sampling Unit 36NV

1. This testing unit contains sodium sulfite as a preservative and caution should be exercised. This unit is used for both chlorinated and unchlorinated samples. Shake unit in upright position to drain the preservative from cap. Do not rinse bottle before collection. Immediately flush skin with large amounts of water if spill occurs.

2. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle.

3. Do not rinse the bottle with sample. Select a clean faucet avoiding such attachments as aerators, dishwasher connectors, etc. Allow water to run for ten minutes at full flow at the sampling point.

4. Reduce flow and collect the sample directly into the bottle. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the bottle. Avoid splashing. Fill bottle to the bottom of the neck. Cap and invert five times to mix sample with the preservative.

Directions for Sampling Unit 36VO

1. These sample vials contain a diluted ascorbic acid preservative and caution should be exercised. Each sample set consists of two bottles. This sampling unit should be used for both chlorinated and unchlorinated samples. Tap each vial while in an upright position to drain the preservative from cap. Do not rinse vials before collection. Immediately flush skin with large amount of water if spill occurs.

2. Do not open the first vial until ready to collect the sample. Do not touch the inside of the cap or vial.

3. Do not rinse the vial with sample. Select a clean faucet removing such attachments as aerators, dishwasher connectors, etc. Allow water to run for ten minutes at full flow at the sampling point.

4. Reduce flow and collect the sample directly into the vial. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the vials. Avoid splashing. Fill the vial until water rounds at the top of vial. Cap and invert to check for air in vial. THE SEPTA (RUBBER PART INSIDE CAP RING) MUST BE SMOOTH SIDE DOWN IN CONTACT WITH SAMPLE - RUBBER SIDE UP TO AVOID POSSIBLE CONTAMINATION.

5. If air is observed in inverted sample, remove cap, add water (DON'T DUMP SAMPLE), and recap as instructed in (4) above.

6. Immediately fill the second vial in the same manner as the first.

Directions for Sampling Unit 36MP

1. These sample vials contain 0.75% hypochlorite solution. Tap each vial in upright position to drain solution from cap. Each sampling unit consists of two vials. Do not rinse the vials before collection.

2. Do not open the vials until ready to collect the sample. Do not touch the inside of caps or vials.

3. Do not rinse the vials with sample. Select a clean faucet removing such attachments as aerators, dishwasher connectors, etc. Allow water to run for ten minutes at full flow at the sampling point.

4. Reduce flow and collect the sample directly into the first vial. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the vial. Avoid splashing. Fill vial until water rounds at the top of vial. Cap and invert to check for air in vial. THE SEPTA (RUBBER PART INSIDE CAP RING) MUST BE SMOOTH SIDE DOWN IN CONTACT WITH SAMPLE - RUBBER SIDE UP TO AVOID POSSIBLE CONTAMINATION.

5. If air is observed in inverted sample, remove cap, add water (DON'T DUMP SAMPLE), and recap as instructed in (4) above.

6. Fill the second vial provided in the same way.

Directions for Sample Unit 36HS

1. Use a field chlorine residual test kit to determine the presence or absence of free chlorine. If free chlorine is absent, proceed with the collection of the sample using Unit 36HS. If a free chlorine residual is present, hydrogen sulfide will not be present.

2. This testing unit contains a diluted zinc acetate preservative in the sample bottle and caution should be exercised. Tap sample bottle in upright position to drain preservatives from cap. Do not rinse bottle before collection. If preservative is spilled, immediately flush skin with large amounts of water.

3. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle.

4. Select a clean faucet avoiding such attachments as aerators, dishwasher connectors, etc. Allow water to run for ten minutes (until cold) at full flow from the sampling tap.

5. Reduce flow to avoid splashing, and collect the sample directly into the bottle. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the bottle. Fill bottle to one-half inch below the bottom of the neck. Cap and invert five times to mix sample with the preservative.

Directions for Sample Unit 36GY

1. Use a field chlorine residual test kit to confirm the absence of a free or combined chlorine residual. If no chlorine residual is present, proceed with the collection of the sample using Unit 36GY. If a free chlorine residual is present, glyphosate will not be present.

2. This testing unit may contain caustic sodium thiosulfate preservatives and caution should be exercised. Tap unit in upright position to drain preservatives from cap. Do not rinse bottle before collection. If preservative is spilled, immediately flush skin with large amounts of water.
3. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle.

4. Select a clean faucet avoiding such attachments as aerators, dishwasher connectors, etc. Allow water to run for ten minutes (or until cold) at full flow at the sampling point.

5. Reduce flow to avoid splashing, and collect the sample directly into the bottle. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the bottle. Fill bottle to the bottom of the neck. Cap and invert five times to mix sample with preservatives.

Sample Submission Forms

A separate submission form, which is included in the appendix, is required for each sample container in order to identify it for reporting. Enter all known information. Type or print legibly using soft lead or waterproof ink. Attach comments regarding testing needs on a separate sheet. The sample forms relay all the important data to the laboratory and users of the analytical results. If the form is not completed properly, the laboratory may delay the analysis or even "dump" the sample. Also, improperly completed paper work may cause monitoring or MCL violations.

The following discussion describes information requested on the sample submission form (left to right, top to bottom) which may not be obvious from the form information:

1. Circle number codes given on submission form for - SAMPLE SOURCE CODE, that best describes water supply or other sample source. SAMPLING PURPOSE CODE, the primary reason for doing the testing. SAMPLE POINT CODE, treatment and location in water supply (other for nonpotable sources).

2. WSSN - Enter WSSN, (Water Supply Serial Number), if sample source code is 1 or 2.

3. Indicate if the sample contains chlorine.

4. REPORT RESULTS AND BILLING TO - Only a single address (Reporting/Billing) can be accommodated for reporting and billing of any testing fees. Use address best able to distribute information to other persons involved.

5. Sample Collector Name - Include first initial after name if more than one person at reporting address has the same last name.

6. Date Collected - Enter the month/day/year when the sample was collected.

7. Time Collected - Enter the time of sample collection to nearest hour and circle AM or PM.

8. Who Collected Sample? - Circle code number from COLLECTOR CODES on form which best describes person collecting the sample.

9. System/Owner Name - Enter name of source (public water system, lake, river, pool, etc.) or name of owner of sample source (homeowner, pool owner, etc.) which best identifies the source.

10. Collection Site - Enter the street address, if applicable. Otherwise describe sampling location, such as Well No. 1. Public water systems may enter Sampling Station Nos.

11. Township, Well-Key - Enter the township name. Public water systems may enter a well-key number

12. Section - Enter standard survey section number if known. Public water systems may disregard this data element.

13. City - Enter the city name of the mailing address for the sampling site.

14. County - Enter the county name for the sampling site.

15. Zip - Enter the zip code for the sampling site.

16. Well Number - For public water systems, enter your well number for the sampling site, if applicable. For all other sampling sites, disregard this data element.

17. Sampling Point - Describe the sampling point, such as kitchen faucet.

18. Site Code - Enter local area site code designation if known. This is generally used to designate sites related to areas under investigation by state/county environmental health programs. Public water systems should consult the state or county health department for this information. All other samplers may disregard this data element.

19. Test Code and Fee - Codes and related test fees are given on the laboratory fee schedule. A partial listing of the schedule is given on the submission form. The full fee schedule is available from state and county health departments. A complete listing of codes is listed in the "Water Analysis Section Testing Catalog".

Note: Private well owners should consult local county or district health department personnel regarding needed testing and proper arrangements to obtain types of testing not given on submission form.

20. Sample Unit # - Enter the Sample Unit Number given on the label of the sample unit used to collect the sample (label will be on bottle used for sample unit). Note that this should match the sample unit numbers required in the fee schedule.

21. Amount Enclosed - If payment of testing fees is submitted with the sample, enter the amount of the check enclosed with the sample (make check payable to State of Michigan). Payment should be made by check or money order. DO NOT SEND CASH. Public water supplies with established charge accounts and Customer Numbers may disregard this data element.

22. Customer Number - Supplies with established charge accounts not submitting payment with samples should enter their unique customer number. All others disregard this data element.

Field Blanks

The use of field (trip) blanks are required by several of the U.S. EPA approved methods for the analysis of organics, such as MDEQ laboratory scans CXTM, CXMP, CXVO, CXEV, and CXPT. Although required by the method, the use of trip blanks will be restricted to situations where there are high probabilities for detecting VOC or SOC. These situations include analyzing samples from locations of known or suspected ground water contamination, inland streams during the peak runoff seasons, or as part of the resampling activity for previously positive results. Also, field blanks will be used as part of the TTHM sampling effort from public water supplies when the results are expected to approach or exceed the MCL.

If field blanks are used and the results from the regular VOC or SOC sample results are negative, the field blank will not be analyzed. On occasion, the field blanks may be analyzed from these sample sets in place of the method blank. When used with TTHM samples, the field blank will only be analyzed when the TTHM results exceed fifty percent of the MCL.

The restricted use of field blanks is due to practicality, economics, and the predictability of sample results from many of the public water supplies. The practicality issue is related to shipping water to and from sample sites. The MDEQ routinely relies on the U.S. Postal Service or other commercial delivery services to ship bottles to many of its users. This delivery method increases the probability of breakage. In addition, many of the local health departments and public water supplies maintain a supply of MDEQ bottles to be used in routine monitoring programs. One of the economic issues is related to the shipping costs of blanks. The main economic issue is the added cost of analysis of samples that yield little useful information. The predictability factor is due to the long monitoring history from the public water supplies, where previous sample results may be used as a guide to speculate on the results from a new sample set.

Also, the restricted use is associated with the reasons field blank analyses were incorporated into the methods' quality control procedures by the U.S. EPA. According to the individuals involved with the development of the U.S. EPA approved methods, field blank analyses were initially required to detect the contamination of TTHM samples by freon leaking from the storage refrigerators. As additional methods were developed, the requirements for the analyses of the field blanks were carried over to the quality control procedures for the VOC due to the similarity of the methods and contaminants. The requirements were added to the SOC quality control procedures without any thought on why the field blank analyses were first required.

The decision to use a field blank will be made by the sample collector. Therefore, the sample collector must order the field blank from the MDEQ laboratory prior to the collection of the samples. When the field blank is received, it must be stored with the routine sample containers. As the name implies, the field blank is transported into the field and shipped backed to the laboratory with the sample containers.

Transporting Samples

Pack samples for shipment using any packaging components supplied with the sample container. If possible, ship sample units together in a single box. Be sure each container has the correct form within (ATTACHED TO OR IN SAME BOX AS SAMPLE BOTTLE).

All bacteriological samples must be delivered directly to the laboratory within thirty hours of collection. The samples may be delivered by the sample collector, a courier, or one of the overnight delivery services. Whenever practical, the samples should be transported in an ice chest or cooler. Frozen samples will not be accepted by the laboratory for analysis. Samples waiting for delivery should be refrigerated.

The transit or holding time for other types of samples varies, as shown in the "Water Analysis Section Testing Catalog". The samples must arrive at the MDEQ laboratory well in advance of the holding time and preferably within twenty four hours of collection. Rapid delivery back to the laboratory is suggested to allow the laboratory a sufficient amount of time to schedule the analysis of the samples. The scheduling of the analysis is exceptionally difficult for the laboratory due to the high volume of samples and variable work load.

For all chemical samples, except for the metals, the U.S. EPA requires the cooling of the sample to 4^0C on the same day as collection. If the samples will be mailed to the laboratory, they must be sent by a guaranteed overnight delivery service on the same day as collection. If the samples will be delivered to the laboratory, it is suggested that the samples be transported to the laboratory in a cooler or ice chest. Prior to transporting the samples, they should be stored in a refrigerator. Coolers are provided for the MDEQ staff to transport the samples to the laboratory.

DEQ

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
ENVIRONMENTAL SCIENCE AND SERVICES DIVISION, DRINKING WATER LABORATORY

REQUISITION FOR WATER SAMPLE UNITS

By authority of PA 368, 1978

ORDER INFORMATION

Name			WSSN(public supply)
Street Address			Date
City	State	Zip code	Area Code & Phone Number

INSTRUCTIONS

- Determine TEST CODE and UNIT NUMBER for desired analysis using the Testing Fee Schedule on reverse side.
- Indicate QUANTITY NEEDED next to appropriate UNIT NUMBER. **DO NOT ORDER MORE SUPPLIES THAN WILL BE USED IN 3 MONTHS.**
- Testing is available only for determining the quality of drinking water, swimming pools and surface water.

UNIT NUMBER	Quantity Of **SINGLES** Needed	Quantity Of **CASES** Needed	**UNIT PURPOSE** (Preservatives - if any)
30			Bacteriological Testing - (10% Sodium Thiosulfate) *40 bottles per case - **limit 2***
30a	*cartons*		Bacteriological Testing - Six bottle carton (no mailing supplies included)
31			Bacteriological & Automated Partial Chemistry - Two Bottles *20 sets per case*
32			Automated Partial Chemistry *40 bottles per case*
32a			Disinfection Byproducts - (0.125mL of 5% Ethylenediamine)
33			Complete Minerals and Corrosion Control
36AC			Ammonia Testing - (0.5mL 1+1 Sulfuric Acid)
36CC			Lead & Copper Testing
36CN			Cyanide Testing, Unchlorinated Sources - (2.0mL of 10N Sodium Hydroxide)
36CNa			Cyanide Testing, Chlorinated Sources - (0.2g of Ascorbic Acid & 1 vial containing 2.0mL 10N Sodium Hydroxide)
36DQ			Diquat & Paraquat Testing - (0.1g sodium thiosulfate)
36EN			Endothall Testing - (0.02g sodium thiosulfate)
36FY			Fluorescent Dye Testing (Charcoal Packets)
36GY			Glyphosate Testing - (0.012g sodium thiosulfate)
36HA			Dalapon & Haloacetic Acids Testing (0.025g ammonium chloride)
36HS			Sulfide Testing - (2.5 mL of Zinc Acetate)
36LP			Carbamate Pesticides, Unchlorinated Sources - (3.6mL Monochloracetic Acid)
36LPa			Carbamate Pesticides, Chlorinated Sources - (0.01g of Sodium Thiosulfate & 1 vial containing 3.6 mL Monochloracetic Acid)
36ME			Metals, Except "first draw" lead copper samples requiring Unit 36CC
36NV			Synthetic organic compounds and Chlorinated Pesticides/Herbicides - (0.05g Sodium Sulfite)
36MP			Trihalomethane Formation Potential - (0.11mL 15:100 bleach) **One Month Expiration Made to Order**
36TO			Total Organic Carbon - (2 drops of 1+1 Phosphoric Acid)
36VO			Volatile Organic Substances - (0.025g Ascorbic Acid) *25 sets per case*
36VOb			Field blank - *14 Day Expiration* - MADE TO ORDER
37			Regulatory Monitoring for Public Water Supplies - includes: 36ME, 36VO, 36NV(2), 36LP, 36CN, 32
38			Unregulated Contaminate Monitoring Rule - includes: 36VO(2), 36NV(3), 32
			Other (please specify)

Orders may be telephoned to (517) 335-8184 or faxed (517) 335-8562.

This requisition may also be mailed to: **WATER SAMPLE UNIT ORDERS**
MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
P O BOX 30270
LANSING MI 48909

DRINKING WATER LABORATORY - **LANSING**
MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

REQUEST FOR WATER ANALYSIS

Issued under authority of Act 368, P.A. 1978

- A form is required for **each sample site** (Collection Site and Sampling Point must be the same for all samples with this form).
- **PREPAYMENT OR CUSTOMER ACCOUNT NUMBER IS REQUIRED FOR TESTING.**
- **Complete all parts of this form which apply.** Samples not properly identified or not having clear test requests MAY NOT be tested.
- Fee amounts are subject to annual changes.

Customer Account Number	**OR**	Check # (payable to **State of Michigan)**	Amount enclosed

WSSN (Type I-II Public Water) or Pool Serial Number	Does sample contain chlorine? ☐ Yes ☐ No

DO NOT SEND CASH!

SAMPLE SOURCE - ***Circle One***

0 - Single Family Dwelling
1 - TYPE I (community, apartment, subdivision, mobile home park, etc., with 25 or more residents year round)
2 - TYPE II (school, industry, restaurant, office, etc., serving 25 or more persons - 60 days or more per year)
3 - TYPE III (all other public supplies, duplex, small office, etc.)
7 - Surface Water (includes bathing beach and waste water discharge)
8 - Swimming pool or Spa
9 - Other

SAMPLING PURPOSE - ***Circle One***

0 - Routine Monitoring
1 - Real Estate Transaction
2 - Repeat Sample
3 - Repair/Construction/New Well
5 - Water Quality Problem
9 - Other

SAMPLE POINT - ***Circle One***

1- Public System Well
2- Public System Surface Water
3- Untreated Public Distribution System
4- Treated Public Distribution System
5 - Untreated Private Well
6 - Treated/Softened Private Well
7 - Pressure Tank/Plant Tap
9 - Other

REPORT RESULTS TO: *PLEASE PRINT*

Name	E-mail address	Sample Collector Name
Mailing Address	Area Code & Phone number	Date Collected
City	State / Zip	Time Collected AM PM

SAMPLE COLLECTION INFORMATION *PLEASE PRINT*

Collector Code ***Circle One*** **0** - County Personnel **1**- Water Supply Operator **2**- MDEQ WD staff **3**- Private Citizen **4** - MDEQ Staff other than WD **5** - MDNR Staff **6**- MDA Staff **9**- Other		**System/Owner Name**	
Collection Site (Street Address)		Township (If known)	Section (If known)
City	County	Zip	Well (If more than one) Number
Sampling Point (kitchen, bath, etc.)		Site Code (If known)	

TESTING REQUEST INFORMATION (REQUIRED)

INSTRUCTIONS: Check box next to Test Code(s) of desired analysis. Check the UNIT # on bottle to ensure you have the REQUIRED UNIT for desired analysis. For other types of testing, enter the TEST CODE, UNIT # (located on the sample bottle) and FEE in the area on the right side of this section. Refer to the full Testing Fee Schedule available from county health departments and DEQ Drinking Water Laboratory for other types of testing. **Fee amounts are subject to annual changes.**

☑	TEST CODE	REQUIRED UNIT #	TEST	FEE
	B	30	Drinking Water Coliforms (Bacteriology)	**$12.00**
	SW	30	Swimming Pool Water Coliform	**$12.00**
	NPEC	30	E. coli for **Surface/Wastewater**	**$15.00**
	N	30	Fecal Coliforms for **Surface/Wastewater**	**$15.00**
	R	32	Automated Partial Chemistry, *including Fluoride, Chloride, Hardness, Nitrate, Nitrite, Sulfate, Sodium and Iron*	**$14.00**
	CAS	36ME	Arsenic	**$16.00**
	CPM1	36ME	Iron, Manganese, Copper and Zinc	**$20.00**
	CPB	36ME	Lead	**$16.00**
	CCUB	36CC	Lead/Copper for corrosion control	**$22.00**
	CXVO	36VO	Organic Solvents Screen	**$90.00**

TEST CODE	UNIT#	FEE
TOTAL OF ALL FEES ⏩		

Submit White Copy With Sample - Keep Yellow Copy For Your Records

For additional information contact your local county health department or the Drinking Water Laboratory, telephone (517) 335-8184 or visit our web site: http://www.michigan.gov/deq

Allow two weeks for results on most testing.

SAMPLE COLLECTION INSTRUCTIONS

UNIT#	INSTRUCTIONS
30	1. **This testing unit contains preservatives in the sample bottle.** Do not rinse the bottle with sample. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle. 2. If not collecting sample from a tap (lake, pool, etc.), plunge bottle mouth down, move in continuous arc down and back up from water, discard top half-inch or to 100 ml line. 3. If using a sample tap, select a clean faucet and remove such attachments as aerators, dishwasher connectors, etc. Allow water to run for about ten minutes (until cold) at full flow from the sampling tap. Reduce flow to avoid splashing, and collect the sample directly into the bottle. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the bottle. **Fill bottle only to the bottom of neck, or to 100 ml line.**
32, 32a 33, 36AC 36CN, 36DQ 36EN, 36GY 36HA, 36HS 36LP, 36ME 36NV	1. **Sample bottle may contain preservative** (refer to unit label on bottle)**.** Do not rinse bottle with sample. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle. 2. Select a clean faucet and remove such attachments as aerators, dishwasher connectors, etc. Allow water to run for about ten minutes (until cold) at full flow from the sampling tap. Reduce flow to avoid splashing, and collect the sample directly into the bottle. Do not use an intermediate container. Do not allow water from the outside surface of the faucet to drip into the bottle. **Fill bottle to the bottom of neck.**
36MP 36TO 36VO	1. **The sample vials contain preservative. Tap each vial in upright position to drain preservatives from cap. Do not rinse vial before collection.** 2. Do not open the vial until ready to collect the sample. Do not touch the inside of cap or vial. Select a clean faucet without attachments or leaking stem. Allow water to run for ten minutes (until cold) at full flow. 3. Reduce flow and collect the sample directly into all vials provided. **Fill vial until water rounds at the top of vial. Cap and invert to check for air in vial. THE SEPTA (RUBBER PART INSIDE CAP RING) MUST BE SMOOTH SIDE DOWN IN CONTACT WITH SAMPLE TO AVOID POSSIBLE CONTAMINATION.** 4. If air is observed in inverted sample, remove cap, add water (**DON'T DUMP SAMPLE)** and recap as instructed.
36CNa 36LPa	1. **Enclosed vial contains dilute preservative and caution should be exercised. This testing unit also contains preservatives in the sample bottle. Tap unit in upright position to drain preservatives from cap. Do not rinse bottle before collection.** 2. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle. 3. Do not rinse the bottle with sample. Select a clean faucet without attachments or leaking stem. Allow water to run for about ten minutes (until cold) at full flow from the sampling tap. 4. Reduce flow to avoid splashing, and collect the sample directly into the bottle. Do not use an intermediate container. **Fill to 1" below top of bottle. Cap and invert 5 times to mix sample with preservatives. Carefully add all preservative in vial to sample bottle. Cap the sample and mix sample. Rinse vial and return.**
36CC	1. Do not open the bottle until ready to collect the sample. Do not touch the inside of cap or bottle. 2. **Sampling point should not have been used for a minimum of six hours prior to sampling.** Do not flush the sample tap before sample collection. 3. Place bottle mouth below tap and turn on water filling bottle to the bottom of the neck.

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
ENVIRONMENTAL SCIENCE & SERVICES DIVISION, DRINKING WATER LABORATORY
http://www.michigan.gov/deq/

Jennifer M. Granholm, Governor

Steven E. Chester, Director

TESTING FEE SCHEDULE

- **This Fee Schedule is effective January 1, 2002.** Fee amounts are subject to annual changes.
- See reverse side for description of sample unit codes and unit ordering information.
- **TEST CODE must be indicated in the TESTING REQUEST INFORMATION section of the form submitted with the sample.**

INORGANIC CHEMISTRY

TEST DESCRIPTION	FEE	UNIT NUMBER(S)	TEST CODE
Automated Partial Chemistry (Fluoride, Chloride, Hardness, Nitrate, Nitrite, Sulfate, Sodium, Iron)	$ 14.00	32	R
Cyanide by distillation/colorimetry (unchlorinated)	$ 25.00	36CN	CCN
Cyanide by distillation/colorimetry (chlorinated)		36CNa	
Hydrogen Sulfide by Titration	$ 30.00	36HS	CCHS
Specific Conductance	$ 12.00	32,33	CCON
Ammonia by Specific Ion Electrode	$ 30.00	36AC	CNH3
pH Determination	$ 13.00	32,33	CPH
Resistance for Demineralized Water	$ 10.00	32,33	CRES
Total Alkalinity as CaCO3	$ 16.00	32,33	CTALK
Silica as SiO2 by Colorimetry	$ 14.00	32,33	CSI
Tannates (Presence/Absence)	$ 11.00	32,33	CTAN
Hydrogen Sulfide (Presence/Absence)	$ 23.00	32	CH2S
Fluorescent Dye (Presence/Absence)	$ 17.00	36FY	CFDY
TBAS Screening	$ 13.00	32	CTBAS
Complete Minerals (TALK, Cl, F, NO3, NO2, SO4, SI, PM2, K, CON, PH, Hardness)	$100.00	33	CMIN
Ortho Phospate by Colorimetry	$ 17.00	32	CPO4
CCON, CTALK, CPO4, Calcium	$ 63.00	33	CORR
Disinfection Byproducts	$ 75.00	32a	CDBP
Perchlorate	$ 75.00	32	CCLO4
Total Organic Carbon	$ 30.00	36TO	CTOC

NOTE: Do not request more than **three test procedures** for each unit 32. Larger units may be used for more extensive requests, ie. unit 33 (more than three procedures).

METALS CHEMISTRY

TEST DESCRIPTION	FEE	UNIT NUMBER(S)	TEST CODE
Aluminum	$ 16.00	36ME	CAL
Antimony	$ 16.00	36ME	CSB
Arsenic	$ 16.00	36ME	CAS
Barium	$ 16.00	36ME	CBA
Beryllium	$ 16.00	36ME	CBE
Cadmium	$ 16.00	36ME	CCD
Chromium	$ 16.00	36ME	CCR
Lead	$ 16.00	36ME	CPB
Lead/Copper for corrosion control	$ 22.00	36CC	CCUB
Mercury	$ 16.00	36ME	CHG
Nickel	$ 16.00	36ME	CNI
Potassium	$ 13.00	32,33	CK
Selenium	$ 16.00	36ME	CSE
Strontium	$ 16.00	36ME	CSR
Thallium	$ 16.00	36ME	CTL
Tin	$ 16.00	36ME	CSN
Calcium/Magnesium/Sodium	$ 18.00	32,33	CPM2
Iron/Manganese/Copper/Zinc	$ 20.00	36ME	CPM1
Complete Metals for Private Wells (AS,SE,BA,CD,CR,HG,PB,PM1)	$ 70.00	36ME	CMET
Complete Metals for Public Supplies (SB,AS BA,BE,CD,CR,HG,PB,SE,NI,TL)	$ 80.00	36ME	CMET2

VOLATILE ORGANIC (VOC) CHEMISTRY

TEST DESCRIPTION	FEE	UNIT NUMBER(S)	TEST CODE
EDB and DBCP by GC/MS	$ 70.00	36VO	CXEV
Max Potential TTHM Formation	$ 55.00	36MP	CXMP
Organic Solvents Screen by ITD	$ 90.00	36VO	CXVO
Trihalomethanes	$ 55.00	36VO	CXTM
p-Dioxane	$115.00	36VO	CXPD

NOTE: Each test procedure requires a **separate sample unit**. Where possible, all detected substances will be identified by mass spectral examination. Names of specific compounds of concern should be provided along with sample and test request.

NON-VOLATILE ORGANIC (SOC) CHEMISTRY

TEST DESCRIPTION	FEE	UNIT NUMBER(S)	TEST CODE
Acidic Phenols by ITD	$150.00	36NV	CXAC
Aromatics by ITD	$110.00	36NV	CXPA
Organophosphate Pesticides by NPD	$150.00	36NV	CXPO
UV Active Pesticides by HPLC/UV	$110.00	36NV	CXLU
Carbamates by HPLC/PCD(unchlorinated)	$ 95.00	36LP	CXLP
Carbamates by HPLC/PCD(chlorinated)		36LPa	
Chlorinated Acid Herbicides	$ 95.00	36NV	CXHB
Pesticides Screening by ECD & NPD	$120.00	36NV	CXPT
Dalapon & Haloacetic Acids	$120.00	36HA	CXHA
Diquat	$150.00	36DQ	CXDQ
Glyphosate	$100.00	36GY	CXGY
Endothall	$150.00	36EN	CXEN

NOTE: Generally each test procedure requires a **separate sample unit**. If more than one type of testing is needed, contact the laboratory to determine the number of different units needed. Where possible, all detected substances will be identified by mass spectral examination. Names of specific compounds of concern should be provided with sample and test request(s).

MICROBIOLOGY

TEST DESCRIPTION	FEE	UNIT NUMBER(S)	TEST CODE
Drinking Water Coliforms(total & E. coli)	$12.00	30,30a	BPTC
Standard Plate Count(48 hours)	$12.00	30,30a	BSPC
Fecal Coliforms for Surface/Wastewater	$15.00	30,30a	NPFC
E. coli for Surface/Wastewater	$15.00	30,30a	NPEC
Swimming Pool Water Coliform	$12.00	30,30a	NSPC
Non-Potable Water Standard Plate Count (pools)	$12.00	30,30a	NPPC

NOTE: Surface water bacteriology is intended only to estimate bacterial group populations.

For questions regarding testing, contact
Drinking Water Laboratory
(517) 335-8184 - Lansing
(906) 482-3011 - Houghton

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

LABORATORY SERVICES SECTION
ENVIRONMENTAL LABORATORY
QUALITY ASSURANCE PROGRAM PLAN

APPENDIX G

Laboratory Certification Program for Chemical and Microbiological Analysis of Drinking Water

QAPP-Ver2.0, Revison 0
May 5, 2003

Laboratory Certification Program For Chemical and Microbiological Analysis of Drinking Water Laboratories

Drinking Water Authority:

The Michigan Safe Drinking Water Act (Act 399, P.A. 1976, as amended) and Administrative Rules promulgated under the Act authorizes the Michigan Department of Environmental Quality to review and certify laboratories used or intended for use in the testing of water from public water supplies for determining compliance with state drinking water standards.

Scope and Applicability:

This certification program is applicable to all laboratories performing analysis of drinking water for the purposes of fulfilling the monitoring and reporting requirements of the rules and regulations promulgated under the Michigan Safe Drinking Water Act, the Federal Safe Drinking Water Act, or any subsequent state act requiring the use of a certified laboratory for drinking water analysis. The program will not prevent uncertified laboratories from performing any quality control or other tests when the state has not required such tests to be performed by a certified laboratory.

Definitions:

Analyst: Any person who performs analysis for certain or all parameters on samples submitted to the environmental laboratory and who meets the qualifications set in this program.

Certification: Official recognition by the Michigan Department of Environmental Quality of a laboratory's ability to perform analysis of public drinking water supply samples under the Safe Drinking Water Act. The recognition is based on the Michigan Department of Environmental Quality's determination that a laboratory has met all the minimum requirements of the Michigan Department of Environmental Quality's policies and procedures, the critical elements in the U.S. EPA Manual for the Certification of Analyzing Drinking Water, April 1990, or any subsequent revised editions of the manual and any requirements published by the U.S. EPA in the Federal Register. Certification is accompanied by a letter of certification and issuance of an official certificate.

Certified Laboratory: A laboratory that has successfully analyzed required performance evaluation samples, passed the on-site inspection, and has been granted certification by the Michigan Department of Environmental Quality.

Certification Officer: Any person designated by the Michigan Department of Environmental Quality to inspect and evaluate environmental laboratories and/or review the laboratory inspection reports generated by the third party for compliance in meeting the criteria set forth in this program.

Consultant: A person who is retained by a written agreement to provide professional consultation services.

Contaminant: The known substance contained in the performance evaluation or environmental sample for which an analysis is performed.

Environmental Laboratory: Any facility that performs analysis on environmental samples to determine the quality of public drinking water supplies, surface water, ground water, and recreational waters.

Laboratory Director: The person who is responsible for the operation of an environmental laboratory and who meets the qualifications set forth in this program.

Laboratory Supervisor: A person who supervises the performance of the analytical procedures within an environmental laboratory and who meets the qualifications set forth in this program.

Major Change in Personnel: The loss or replacement of the laboratory supervisor or a situation in which a trained and experienced analyst is no longer available to analyze a particular contaminant for which certification has been granted.

Major Remodeling: Remodeling of the laboratory facility that requires the acquisition of a local building permit.

Maximum Contaminant Level (MCL): A maximum permissible concentration of a contaminant in drinking water.

MDEQ: The Michigan Department of Environmental Quality

MI SDWA: The Michigan Safe Drinking Water Act (Act 399, P.A. 1976, as amended)

Noncertified Laboratory: A laboratory that possesses major deficiencies and/or cannot produce valid data as determined by the Michigan Department of Environmental Quality.

Performance Evaluation (PE) Samples: Samples that include contaminants in known concentrations (unknown to the laboratory), which are provided to laboratories for analysis to determine if the laboratory can satisfactorily perform specific analysis.

Proficiency Testing: Laboratory testing and analysis of performance evaluation and/or quality control samples provided by the Michigan Department of Environmental Quality, the third party, or other sources.

Program Manager: Designated third party representative responsible for implementing and operating the program. This individual will be responsible for on-site inspections, liaison with the Michigan Department of Environmental Quality, and consulting with certified laboratories.

Probationary Certification: A certification status granted by the Michigan Department of Environmental Quality to an environmental laboratory, which has corrected all deficiencies, but historically has several minor problems with personnel or practices.

Provisionally Certified Laboratory: A laboratory that has deficiencies, but can still produce valid data as determined by the Michigan Department of Environmental Quality.

Public Water Supply: All mains, pipes, and structures through which water is obtained and distributed to the public, including wells and well structures, intakes and cribs, pumping stations, treatment plants, reservoirs, storage tanks and appurtenances, collectively or severally, actually used or intended for use for the purpose of furnishing water for drinking or general domestic use and which serve at least 15 service connections or which regularly serve at least 25 persons at least 60 days per year.

Quality Control (QC) Samples: Water samples, which include contaminants in exact concentrations (known to the laboratory), provided to laboratories to assist them in evaluating their own performance.

Reciprocity: Mutual acceptance of a certification between the Michigan Department of Environmental Quality and other primary enforcement authorities of the Federal Safe Drinking Water Act.

SDWA: The Safe Drinking Water Act (Act 399, P.A. 1976, as amended)

Third Party: The organization(s) independent of the Michigan Department of Environmental Quality and environmental laboratories with whom the Michigan Department of Environmental Quality has a working agreement or contract for the field inspection and evaluation of environmental laboratories.

U.S. EPA: United States Environmental Protection Agency.

Certification Procedures:

A. An environmental laboratory which meets or exceeds the minimum criteria for certification may receive certification from the MDEQ for any organic chemical, inorganic chemical, or microbiological parameters for which methodologies have been specified by the drinking water regulations (40 CFR 141.21-141.25) or otherwise approved by the U.S. EPA for compliance with the SDWA.

B. The operational aspects of an environmental laboratory that will be evaluated in considering certification are:
   1. Physical facilities.
   2. Personnel.
   3. Methodology and instrumentation.
   4. Data handling.
   5. Quality control and assurance.
   6. Results of proficiency tests.

C. In seeking certification, the petitioning environmental laboratory must:
   1. Submit a formal request for certification to the MDEQ.
   2. Complete any applicable questionnaires furnished by the MDEQ giving information listed in Part B.
      a. Respond in a timely fashion to any questionnaire or inquiries from the third party or the MDEQ dealing with the operation, maintenance, or other data related to the certification of the environmental laboratory.
         1.) Environmental laboratories may be given a specified period of time to return the certification application, fee payments, and any other supporting/requested documentation.
         2.) The typical period of time for the environmental laboratories to return the requested material will be 90 days.
         3.) The time period will commence from the date the material is submitted to the environmental laboratory.
         4.) Based on a written request from the environmental laboratory, the certification officer may grant an extension to the response period.
   3. Analyze PE samples and possibly QC samples provided by the third party, the MDEQ, or the U.S. EPA, and report the results of the analysis to the MDEQ and/or the U.S. EPA.
   4. Permit an on-site inspection of the environmental laboratory by the third party or the MDEQ or a joint inspection by the third party and the MDEQ. The on-site inspection(s) will be arranged at the mutual convenience of all parties.
      a. The certification inspection of the environmental laboratory shall be scheduled to be held within 90 days after the application and fee payment has been returned to the MDEQ.

b. A written report will be submitted to the environmental laboratory from either the third party or the certification officer within 30 days following the inspection.
   1.) The written report shall state variances from the practices and requirements specified in other sections of this program which were found during the inspection or subsequent investigations.
   2.) The written report may request the environmental laboratory to implement certain actions to correct the variances.
   3.) The environmental laboratory must submit a written response regarding plans to implement any corrective measures requested in the report within 30 days of receiving the report.
   4.) The certification officer may base any decisions on the certification status of the environmental laboratory based on available information on file 30 days after the written report is submitted to the environmental laboratory.

5. Pay all fees associated with the certification process.
   a. Payment of fees for the certification inspection shall be made at the time the application is submitted to the MDEQ and prior to the on-site inspection.
   b. Environmental laboratories, where applicable, will be required to pay all fees associated with reagent water testing or obtaining proficiency samples.
   c. Fee schedules shall be formally established for all areas of the certification program.
      1.) The certification inspection and reagent water testing fee schedule and any subsequent adjustments shall be established by the state legislature. The proficiency sample fee schedule and method of payment shall be equivalent to the procedures established by the U.S. EPA or its designated contractor.

D. After the procedures in Part C have been completed, the MDEQ will:
1. Certify the environmental laboratory if appropriate.
   a. Certification will be based on the MDEQ review of the third party's or the MDEQ's inspection of the environmental laboratory.
   b. Certification will be based on the MDEQ review of any available information including information generated in Parts B and C.
   c. Certification will be effective for a three year period from the date of issue unless modified or revoked by the MDEQ.
   d. Certification will not be transferable. In the event of change of ownership, director, supervisor, analysts, or relocation or major remodeling of the physical plant of the environmental laboratory, the MDEQ and the third party must be notified in writing within 30 days.

e. Certification will be contingent upon a satisfactory quality control inspection by the third party or the MDEQ during the certification period.
   1.) Quality control inspection may or may not be made during certification period for any individual environmental laboratory.
   2.) Quality control inspection may be done randomly for all certified environmental laboratories.
   3.) Quality control inspection may be done at an individual environmental laboratory where either the third party or MDEQ suspects or has previously noted a deficiency, or as a result of a complaint.
   4.) In general, approximately 10% of all laboratory inspections performed will be quality control inspections.

2. Deny certification of the environmental laboratory.
   a. Denial of certification will be in writing.
   b. Denial of certification will list reason(s) for action.
   c. Denial of certification will include the third party or the MDEQ inspection report and information as to corrective action(s) needed.
   d. Denial of certification will not prevent the environmental laboratory from:
      1.) Reapplying for certification after corrective measures have been taken.
      2.) Seeking consultation from the MDEQ or the third party.
      3.) Performing any tests when the state has not required such tests to be performed by a certified laboratory.

3. Provisionally certify the environmental laboratory.
   a. May be issued when deviations or deficiencies from the applicable requirements are found, but the MDEQ believes the environmental laboratory can still generate acceptable results.
   b. May be issued as a result of normal certification inspection by the third party or the MDEQ.
   c. May be issued based on analytical results from any PE or QC samples.
   d. May be issued to a certified environmental laboratory as a result of a special quality control inspection performed during the normal certification period.
   e. Provisional certification may be effective up to 12 months from the date of issue unless modified, revoked, or upgraded by the MDEQ.
      1.) During the 12 month period of certification, the environmental laboratory's corrective action(s) will be reviewed.
         a.) A one-time extension of six months may be granted by the MDEQ for uncorrected deficiencies that the environmental laboratory is making a good faith effort to resolve.
         b.) If no effort to resolve the deficiencies has been made, certification will be revoked.

2.) The MDEQ may elect to keep a laboratory on provisional status after acceptable corrective action has been made, until the corrections are confirmed by an on-site visit by the third party, or MDEQ.

f. Provisional certification will not prevent the environmental laboratory from:
   1.) Reapplying for full certification after corrective measures have been taken.
   2.) Performing any tests required by the state to be performed by a certified laboratory unless restricted by the MDEQ when the provisional certificate was issued.
   3.) Seeking consultation from the MDEQ or the third party.

4. Probationary certification of the environmental laboratory.
   a. May be issued to an environmental laboratory, which has corrected all deficiencies, but historically has several problems with personnel or practices.
      1.) Probationary certification will typically be used by the MDEQ to express its concerns to an environmental laboratory regarding repeated or series of different deficiencies from one inspection to the next inspection.
      2.) Probationary certification may be used by the MDEQ to express its concerns to an environmental laboratory regarding the serious nature of a single major deficiency, which was found in a single inspection and has been corrected.
   b. Environmental laboratories issued probationary certification will receive closer scrutiny from the MDEQ or the third party.
      1.) Closer scrutiny may include special quality control inspections or special requests for information.
      2.) Closer scrutiny may include requests for the environmental laboratory to analyze additional PE samples.
   c. A probationally certified environmental laboratory has the full rights and responsibilities of a fully certified laboratory.
   d. An environmental laboratory may request upgrading to full certification after it has demonstrated its ability to maintain a deficient free facility.
      1.) The time period required for a laboratory to remain at the probationary rating will vary with the nature of the past deficiencies.
      2.) The environmental laboratory may request an explanation of the MDEQ's reasons for the probationary rating and the duration of the rating.
   e. May be issued to an environmental laboratory, which has no known current or past deficiencies.
      1.) Issuance of probationary certification under these circumstances will be done when the U.S. EPA mandates the use of restricted, conditional, or temporary certificates.

2.) Issuance may be done for any contaminant, group of contaminants, or analytical technique which is new to the environmental laboratory, or recently regulated contaminant.
   a.) Successful analysis of a set of PE samples may be required prior to certification.
   b.) Certification may be done at the request of the environmental laboratory.
   c.) Certification may be administratively issued by the MDEQ to an environmental laboratory which holds certification for similar contaminants prior to a formal on-site inspection.

f. Probationary certification will be effective for a three year period from the date of issue unless modified or revoked by the MDEQ.

Conditions of Certification

A. The environmental laboratory shall notify the third party and the Michigan Department of Environmental Quality of major changes.
   1. Major changes are considered to be:
      a. Major remodeling.
      b. Major change in personnel.
      c. Changes of laboratory director, supervisor, or owner.
      d. Relocation of the environmental laboratory.
      e. New laboratory equipment and/or changes of analytical procedures.
   2. As a result of any major changes the third party, or the MDEQ may:
      a. Request analysis of PE or QC samples.
      b. Request resumes of new personnel, director, supervisor, or owner.
      c. Make an on-site visit:
         1.) Visit may be informal.
         2.) Visit may be special quality control inspection.

B. The MDEQ may revoke or downgrade certification status. Any of the following may be cause for revocation of certification or downgrading the certification status:
   1. Failure to pass a regular certification or quality control inspection.
   2. Unsatisfactory analyses of PE or QC samples.
   3. Failure to notify the MDEQ of major changes within 30 days.
   4. Fraud or other criminal activity.
   5. Falsification of records or analytical results.
   6. Failure to respond to any MDEQ inquiry or questionnaire.
   7. Voluntary cancellation of certification.

C. The MDEQ will take several factors into account in considering actions to be taken against certified, probationally certified, or provisionally certified environmental

laboratory where deviations or deficiencies have been found. The factors include but are not limited to the following:

1. The length of time the deviation has existed.
2. The environmental laboratory's prior record of deviations and deficiencies.
3. The environmental laboratory's prior record of response in correcting deviations and deficiencies.
4. Whether the environmental laboratory knowingly caused or allowed the deviation or deficiencies.
5. The potential effect of the deviation or deficiency on the quality of the analytical data generated by the environmental laboratory.

D. Certification will be limited to the analytical procedures for which an environmental laboratory has been approved.

1. All contaminants for which the environmental laboratory has been approved will be listed on the certificate of approval.
   a. Certification will include contaminants for which compliance monitoring is mandated by the U.S. EPA or the MDEQ.
      1.) Certification will include all the regulated contaminants.
      2.) Certification may include the unregulated contaminants.
   b. Certification will not include contaminants for which compliance monitoring is not required by the U.S. EPA or MDEQ.
2. All procedures for which the environmental laboratory has been approved will be specified in writing.
3. The environmental laboratory shall not claim in any fashion that it is certified to perform any type of analysis for which it lacks the proper certification.
4. The MDEQ may elect to certify an environmental laboratory for a general analytical technique or group of contaminants versus individual parameters.
   a. The certification for a general analytical technique or a group of contaminants will be in writing.
   b. All individual contaminants accepted under the certified technique will be in writing.
   c. The certification for a general technique may be for the successful analysis of all or part of the PE or QC samples for the individual contaminants listed under the technique.
   d. The certification for partial successful analysis of the contaminants listed for a general technique will only be done within the guidelines developed by the U.S. EPA.
   e. The MDEQ may require the environmental laboratory to have been previously certified, or concurrently seeking, or to maintain certification for certain contaminants related to the general technique.
      1.) The MDEQ may require certification for total trihalomethanes as a prerequisite for the certification for the regulated volatile organic chemicals.

2.) The MDEQ may require certification for the regulated volatile organic chemicals as a prerequisite for the certification of the unregulated volatile organic chemicals.

3.) The MDEQ may require certification for other regulated chemicals as a prerequisite for certification of unregulated chemicals of the same general classification.

E. The environmental laboratory at its option may display any certificate of approval granted by the MDEQ.

1. The environmental laboratory may not claim that certification by the MDEQ is a guarantee of the validity of data generated by the environmental laboratory.
2. The environmental laboratory may not claim that certification by the MDEQ implies data generated by the environmental laboratory is superior to the data generated by another certified environmental laboratory.

F. An environmental laboratory will be required to analyze PE and possibly QC samples for initial certification or to maintain certification.

1. The environmental laboratory will be required to report the test results to the MDEQ within 30 days of receipt of the PE or QC samples.
2. Samples may be submitted to the environmental laboratory by the third party or MDEQ.
3. Samples may be submitted to the environmental laboratory by the U.S. EPA at the request of the third party or MDEQ.
4. There will be no charge to the third party or MDEQ by the environmental laboratory for the proficiency testing.
5. Failure to provide results within satisfactory precision and accuracy in PE or QC samples may be considered cause for action by the MDEQ.
   a. Action may include quality control or informal inspection by the third party and/or the MDEQ.
   b. Action may include revocation or downgrading of certification for the parameters not within satisfactory limits.
      1.) Failure of one set of PE or QC samples may be cause for the downgrading of certification.
      2.) Failure of two consecutive PE or QC samples may be cause for revocation of certification.
   c. Action may require the environmental laboratory to change equipment procedures or analysts.
   d. Action may include the submission of additional PE or QC samples.
      1.) A waiting period may be required before additional samples are submitted to the environmental laboratory.
      2.) Written assurance to the MDEQ may be required on the corrective measures undertaken by the environmental laboratory.

e. All environmental laboratories must successfully analyze at least one set of PE samples each year and one set of QC samples each quarter.
   1.) Failure to analyze either the PE or QC samples at the prescribed frequency will result in either the downgrading or revocation of the environmental laboratory's certification status.
   2.) The MDEQ may waive the requirement for the environmental laboratory to analyze PE or QC samples when the samples are not available and at the discretion of the U.S. EPA.
   3.) It is the responsibility of the environmental laboratory to submit to the MDEQ the results of the PE samples on at least an annual basis.

G. The environmental laboratory may subcontract with another environmental laboratory.
   1. The name of the environmental laboratory performing the analyses must appear on all reports of analytical results.
   2. For tests that are required to be performed by a certified laboratory, only laboratories with the proper certification may be used as a subcontractor.
   3. Any sample submitted to the environmental laboratory for proficiency testing may not be resubmitted to a subcontractor.

Authority of Certification Officer

The certification officer will have the following authority with this program in dealing with environmental laboratories:

A. To conduct on-site visits at the environmental laboratories.
   1. The third party will be primarily responsible for conducting all on-site visits.
   2. The certification officer may conduct an on-site visit.
      a. Most of the certification officer's on-site visits are expected to be as a part of a joint inspection with the third party.
      b. On-site visits may be considered a part of a certification inspection, a part or an entire quality control inspection, or an informal visit for consultation.
      c. On-site visits by the certification officer may be as a result of a request by the third party, the environmental laboratory, or other interested parties.
      d. On-site visits may be made to verify correction of deviations noted in any inspection report.

B. To act as the MDEQ's liaison with the third party.

C. To review reports generated by the third party regarding the environmental laboratory.

D. To require information relevant to the technical operation of the environmental laboratory.

1. The third party will be primarily responsible for the collection of the information.
2. The certification officer may contact either the environmental laboratory or the third party for the information.

E. To inspect quality assurance records and other pertinent records.

F. To observe and question analysts and work on parameters for which certification is sought.

G. To submit oral and written reports for granting or denying certification based on the completion of the evaluation process.

H. To require the environmental laboratory to analyze and report the results of any PE and QC samples for parameters that the environmental laboratory is certified or seeking certification to the MDEQ or the third party.

Reciprocity Agreements

The MDEQ may enter into agreements with the units of government of other states with SDWA primacy for the recognition of their environmental laboratory inspection or certification.

A. The MDEQ may elect to accept the certification of all environmental laboratories within the jurisdiction of another state.

B. The MDEQ may elect to review the inspection reports and other pertinent data generated by the primacy agency of another state for each individual environmental laboratory located outside of Michigan.

C. The MDEQ may elect to require the environmental laboratories located outside of Michigan to be inspected by the third party.

Oversight

A. The MDEQ will periodically meet with the third party.

1. Progress and criteria of laboratory certification process may be reviewed.
2. Corrections required of certain environmental laboratories may be reviewed.
3. Compliance with the MDEQ-third party contract for conducting the program may be reviewed.

B. The MDEQ will review certain laboratories on-site to determine that corrections to noted deficiencies are being made.

C. The MDEQ will conduct joint inspections of environmental laboratories with the third party from time to time to directly observe the inspection program.

D. MDEQ will investigate complaints of either the third party certification inspection program staff or the environmental laboratories.

Hearings and Appeals

A. The MDEQ will give written notice to the director or owner of an environmental laboratory on any action on revocation, partial revocation, or downgrading of the laboratory's certification.
   1. The notice will include the facts used by the MDEQ for the action.
   2. The notice will include the procedure for requesting a hearing.
   3. The notice may immediately revoke, partially revoke, or downgrade the certification of any environmental laboratory.
      a. Immediate and summary actions will be taken if there is substantial risks to the public health or safety due to laboratory deficiencies compromising the analytical results.
      b. Other actions will allow the environmental laboratory 30 days to respond to the notice.
      c. Immediate and summary actions will be based on documented evidence by the MDEQ.

B. Any hearing requested by an environmental laboratory in response to a notice from the MDEQ will be in accordance with the Michigan Administrative Procedures Act (Act 306, P.A. 1969, as amended).

Criteria for Conducting On-Site Evaluations

A. The third party will be primarily responsible for conducting the on-site evaluations based on the criteria developed by the MDEQ or jointly developed by the MDEQ and third party.
   1. The criteria developed by the third party will be subject to review and approval by the MDEQ.
   2. The criteria used by the MDEQ or the third party must, at a minimum, comply with the provisions of the U.S. EPA Manual for the Certification of Laboratories Analyzing Drinking Water, April 1990, or any subsequent revised editions of the manual, 40 CFR, Part 141 as amended, any directives issued on certification, QA/QC and analytical methodology by the MDEQ or the U.S. EPA, or published in the Federal Register by the U.S. EPA.
   3. The third party will make revisions to their criteria as amendments/revisions are made to the documents listed in A.2. above.

4. The latest U.S. EPA approved edition of Standard Methods may be used in developing the criteria.
5. In the event an MDEQ and third party contract is not in effect, the MDEQ will conduct all inspections.

B. Alternative Test Procedures (ATP).
1. Applicants who propose an ATP on a limited basis need to provide a step-by-step procedure, applicable literature citations or other references, and any available comparability data between the proposed ATP and the U.S. EPA-approved test procedure for the same contaminant.
2. In the case of new techniques where precision and accuracy data may not be available to support an application, the applicant for a limited use ATP will be asked to provide comparability data from the analysis of samples collected from one to five water supply systems most representative of those routinely analyzed. From each of these systems, three samples, in which the concentrations range from the limit of detection (LD) to the maximum contaminant level (MCL), should be collected and each sample analyzed eight times, four times each, by the proposed technique and the U.S. EPA-approved test procedures. Samples can be spiked as necessary to cover the concentration range between the LD and MCL.
3. Applicants will forward their ATP request to the MDEQ. The MDEQ will forward ATP requests for limited use with their recommendations to the Regional Administrator, Region 5, for approval. The Regional Administrator has final authority to approve limited use ATP request.

Reagent Water Testing

A. Laboratory pure water used in the microbiological analyses of drinking water shall have these characteristics:

Property	Value
pH	5.5-7.5
Conductivity	Less than 2.0 micromhos/cm (resistivity greater than 0.5 megohm cm) $\pm$ 1 percent at 25°C
Trace Metals:	
Individual Metals	Less than or equal to 0.05 mg/l
Total Metals (Pb, Cd, Cr, Cu, Ni, Zn)	Less than or equal to 0.1 mg/l
Test for Bactericidal Properties of Distilled Water	Ratio of 0.8 to 3.0

Free Chlorine Residual	None
Heterotrophic Plate Count	Less than 500 CFU's/ml

B. Laboratory pure water shall be analyzed annually by the test for bacteriological quality of distilled water as specified in the latest U.S. EPA approved edition of "Standard Methods for the Examination of Water and Wastewater" or the U.S. EPA's Manual for the Certification of Laboratories Analyzing Drinking Water, April 1990. Only satisfactorily tested water shall be used in preparing media, reagents, rinse, and dilution water. If the water tested does not meet the requirements, corrective action shall be taken and the water retested.

C. Laboratory pure water shall be analyzed monthly for conductance, pH, chlorine residual, and heterotrophic plate count. Heterotrophic plate counts shall be performed as specified in the latest U.S. EPA approved edition of "Standard Methods for the Examination of Water and Wastewater". If the water tested exceeds requirement for these properties, corrective action shall be taken and the water retested.

D. Laboratory pure water shall not be in contact with heavy metals, and shall be analyzed initially and annually thereafter for trace metals (especially Pb, Cd, Cr, Cu, Ni, and Zn). If the water tested exceeds requirements for trace metals, corrective action shall be taken and the water retested.

E. The following quality control tests for heterotrophic plate count shall be utilized:
   1. Sterility controls shall be poured for each bottle of sterile, melted, tempered medium used.
   2. Sterility of pipettes and petri dishes shall be determined.
   3. Microbial density of the air during plating procedures shall be determined for each series of samples plated. When 15 or more colonies appear on an exposed plate after a 15 minute exposure period and 48 hours of incubation at 35°C, corrective action shall be taken.

Response to Analytical Results

A. The environmental laboratory must notify the individual in charge of a public water supply when analytical results of a sample from that supply exceed a maximum contaminant level (MCL).
   1. Notification shall be made as soon as possible.
   2. Notification shall be made on any positive presumptive microbiological results.
      a. Positive presumptive results shall be listed as preliminary.
      b. Verified/confirmed results shall be reported to the public water supply.

c. The purpose of this requirement is to allow the public water supply to begin preliminary investigations and the collection of any check or resamples samples as soon as possible.

3. The initial notification may be made verbally.
4. A final written report shall be made in a timely fashion.

B. The MDEQ may request a copy of the analytical results of any sample from a public water supply.
   1. The results shall be provided in a timely fashion to the MDEQ.
   2. Any analytical results obtained by the MDEQ are open to the general public through the Michigan Freedom of Information Act.

ADDENDUM TO APPENDIX I OF THE MDEQ WATER SUPPLY SAFETY QUALITY ASSURANCE PROGRAM PLAN (EFFECTIVE MARCH 31, 2000)

Issuance of Administrative Extension

Issuance of Administrative Extension (Effective March 31, 2000):

A. In order to allow enough lead-time for a laboratory to be recertified, the Laboratory Certification Program Officer will send out the recertification package seven months in advance of a laboratory's certification expiration date. This lead-time allows for the following windows:
   1. If a third party auditor is to be used to conduct the audit:
      a. The laboratory must return the application package to the State within 30 days.
      b. The State must forward the certification package to the third party auditor within seven days.
      c. The third party auditor must conduct the inspection within 90 days.
      d. The third party auditor must issue an inspection report to the laboratory within 30 days.
      e. The laboratory must provide corrective action plans for the deficiencies cited in the report within 30 days.
      f. The third party auditor must evaluate the corrective actions and make a recommendation regarding the certification status of the inspected laboratory to the State within 15 days.
      g. The State must make a final determination regarding the certification status of the inspected laboratory within seven days.
   2. If the State is to conduct the audit:
      a. The lab must return the inspection package to the State within 30 days.
      b. The State must conduct the inspection within 45 days.
      c. The State must issue a report to the inspected laboratory within 15 days.
      d. The laboratory must provide corrective action plans for the deficiencies cited in the report within 30 days.
      e. The State must evaluate the corrective actions and make a final determination regarding the certification status of the inspected laboratory within 15 days.

B. Occasionally, there may be circumstances beyond the inspected laboratory's control that prevent the laboratory from finishing the recertification process prior to the expiration of their certification. In these cases, the laboratory will be issued an administrative extension of their certification so that they may continue to operate as a certified laboratory until the recertification process can be completed.

C. If an administrative extension is granted, documentation must be maintained in the laboratory's file indicating the reason for the extension. The extension shall be initially given for a period of no more than 90 days.

The laboratory may be issued another 90-day extension after the initial extension, provided that the reason for the extension is documented. The second extension may only be given in extenuating circumstances. Such circumstances include a failure on the part of the third party auditor to fulfill their contractual obligations or a failure on the part of the State to administer the time frame requirements.

D. The certification of the laboratory will lapse if the delay in the recertification process is due to a failure on the part of the laboratory to adhere to the specified deadlines. In these cases, the laboratory will not be considered certified and will be required to begin the certification process as if it were a new laboratory.

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

LABORATORY SERVICES SECTION
ENVIRONMENTAL LABORATORY
QUALITY ASSURANCE PROGRAM PLAN

TABLE 1

MDEQ Environmental Laboratory Capabilities and Reporting Limits

QAPP-Ver2.0, Revison 0
May 5, 2003

Table 1: ENVIRONMENTAL REPORTING LIMITS (RL) FOR DEQ-DWRP LABORATORY SECTION

VOLATILE ORGANICS Method 624/8260	Water/TCLP/SPLP Reporting Limits (ug/L)	MeOH Sediment/Soil Reporting Limits (ug/Kg)	Oil Reporting Limits (mg/Kg)	Water Containers & Hold Times	Sediment/Soil Containers & Hold Times	Oil Containers
1,1,1,2-Tetrachloroethane	1.0	50	1.0	2 - 40mL glass vial	40mL tared glass	5mL of sample
1,1,1-Trichloroethane	1.0	50	1.0	w/Teflon Septum	vial	in glass
1,1,2,2-Tetrachloroethane	1.0	50	1.0	seal	10grams sample	"
1,1,2-Trichloroethane	1.0	50	1.0	14 days HT	in 10mL MeOH	"
1,1-Dichloroethane	1.0	50	1.0	"	w/teflon seal	"
1,1-Dichloroethylene	1.0	50	1.0	"	14 days HT	"
1,2,3-Trichlorobenzene	5.0	250	5.0	"	"	"
1,2,3-Trichloropropane	1.0	50	1.0	"	"	"
1,2,4-Trichlorobenzene	5.0	250	5.0	"	"	"
1,2,4-Trimethylbenzene	1.0	50	1.0	"	"	"
1,2-Dibromo-3-chloropropane	5.0	250	5.0	"	"	"
1,2-Dibromoethane	1.0	50	1.0	"	"	"
1,2-Dichlorobenzene	1.0	50	1.0	"	"	"
1,2-Dichloroethane	1.0	50	1.0	"	"	"
1,2-Dichloroethylene (cis)	1.0	50	1.0	"	"	"
1,2-Dichloroethylene (trans	1.0	50	1.0	"	"	"
1,2-Dichloropropane	1.0	50	1.0	"	"	"
1,3,5-Trimethylbenzene(Mesitylene	1.0	50	1.0	"	"	"
1,3-Dichlorobenzene	1.0	50	1.0	"	"	"
1,3-Dichloropropene (cis)	1.0	50	1.0	"	"	"
1,3-Dichloropropene (trans	1.0	50	1.0	"	"	"
1,4-Dichloro-2-butene(trans)	5.0	250	5.0	"	"	"
1,4-Dichlorobenzene	1.0	50	1.0	"	"	"
2-Butanone (MEK)	5.0	250	5.0	"	"	"
2-Hexanone	5.0	250	5.0	"	"	"
2-Methylnaphthalene	5.0	250	5.0	"	"	"
Acetone (2-Propanone)	20	1,000	20	"	"	"
4-Methyl-2-Pentanone (MIBK)	5.0	250	5.0	"	"	"
Acrylonitrile	5.0	250	5.0	"	"	"
Benzene	1.0	50	1.0	"	"	"
Bromobenzene	1.0	50	1.0	"	"	"
Bromochloromethane	1.0	50	1.0	"	"	"
Bromodichloromethane	1.0	50	1.0	"	"	"
Bromoform	1.0	50	1.0	"	"	"
Bromomethane	5.0	250	5.0	"	"	"
Carbon disulfide	1.0	50	1.0	"	"	"
Carbon tetrachloride	1.0	50	1.0	"	"	"
Chlorobenzene	1.0	50	1.0	"	"	"
Chloroethane	5.0	250	5.0	"	"	"
Chloroform	1.0	50	1.0	"	"	"
Chloromethane	5.0	250	5.0	"	"	"
Dibromochloromethane	1.0	50	1.0	"	"	"
Dibromomethane	1.0	50	1.0	"	"	"
Dichlorodifluoromethane	5.0	250	5.0	"	"	"
Diethyl ether	5.0	250	5.0	"	"	"
Ethylbenzene	1.0	50	1.0	"	"	"
Hexachloroethane	5.0	250	5.0	"	"	"
Isopropylbenzene	1.0	50	1.0	"	"	"
m&p-Xylene	2.0	100	2.0	"	"	"
Methyl Iodide (Iodomethane)	1.0	50	1.0	"	"	"
Methyl Tertiary Butyl Ether (MTBE)	1.0	50	1.0	"	"	"
Methylene chloride	5.0	250	5.0	"	"	"
Naphthalene	5.0	250	5.0	"	"	"
n-Butylbenzene	1.0	50	1.0	"	"	"
n-Propylbenzene	1.0	50	1.0	"	"	"
o-Xylene	1.0	50	1.0	"	"	"
p-Isopropyl Toluene (p-Cymene	1.0	50	1.0	"	"	"
sec-Butylbenzene	1.0	50	1.0	"	"	"
Styrene	1.0	50	1.0	"	"	"
tert- Butylbenzene	1.0	50	1.0	"	"	"
Tetrachloroethylene	1.0	50	1.0	"	"	"
Tetrahydrofurar	5.0	250	5.0	"	"	"
Toluene	1.0	50	1.0	"	"	"
Trichloroethylene	1.0	50	1.0	"	"	"
Trichlorofluoromethane	1.0	50	1.0	"	"	"
Vinyl chloride	1.0	50(40)*	1.0	"	"	"

*Estimated results between 40 and 50

Table 1: ENVIRONMENTAL REPORTING LIMITS (RL) FOR DEQ-DWRP LABORATORY SECTION

BTEX/MTBE VOLATILES Method 624/8260	Water/TCLP/SPLP Reporting Limits (ug/L)	MeOH Sediment/Soi Reporting Limits (ug/Kg)	Oil Reporting Limits (mg/Kg)	Water Containers & Hold Times	Sediment/Soi Containers & Hold Times	Oil Containers
Benzene	1.0	50	1.0	2 - 40mL glass vial	40mL tared glass	5mL of sample
Toluene	1.0	50	1.0	w/Teflon Septum	vial	in glass
Ethylbenzene	1.0	50	1.0	seal	10grams sample	"
m & p-Xylene	2.0	100	2.0	14 days HT	in 10mL MeOH	"
0 -Xylene	1.0	50	1.0	"	w/teflon sea	"
Methyl Tertiary Butyl Ethe	1.0	50	1.0	"	14 days HT	"

Pesticides Method 608/8081	Water Reporting Limits (ug/L)	Sediment/Soi Reporting Limits (ug/Kg)	Oil Reporting Limits (mg/Kg)	Water Containers & Hold Times	Sediment/Soi Containers & Hold Times	Oil Containers
Aldrin	0.02	20	0.2	2 - 1000mL glass	1 - 8 ounce glass	10mL in glass
a-BHC	0.02	20	0.2	amber bottles	14 days HT	"
b-BHC	0.02	20	0.2	7 days HT	"	"
d-BHC	0.02	20	0.2	"	"	"
g-BHC (lindane)	0.02	20	0.2	"	"	"
BP-6 (PPB)	0.05	250	0.7	"	"	"
a-Chlordane	0.02	50	0.2	"	"	"
g-Chlordane	0.02	50	0.2	"	"	"
4,4'-DDD	0.02	20	0.2	"	"	"
4,4'-DDE	0.02	20	0.2	"	"	"
4,4'-DDT	0.02	20	0.2	"	"	"
Dieldrin	0.02	20	0.2	"	"	"
Endosulfan I	0.02	20	0.2	"	"	"
Endosulfan II	0.05	20	0.4	"	"	"
Endosulfan Sulfate	0.05	20	0.4	"	"	"
Endrin	0.02	20	0.2	"	"	"
Endrin Aldehyde	0.05	20	0.4	"	"	"
Endrin Ketone	0.05	20	0.4	"	"	"
Heptachlor	0.02	20	0.4	"	"	"
Heptachlor epoxide	0.02	20	0.4	"	"	"
Hexabromobenzene	0.02	100	0.2	"	"	"
Methoxychlor	0.05	50	0.4	"	"	"
Mirex	0.02	50	0.2	"	"	"
Toxaphene	0.1	170	10	"	"	"

PCBs Method 608/8082	Water Reporting Limits (ug/L)	Sediment/Soi Reporting Limits (ug/Kg)	Oil Reporting Limits (mg/Kg)	Water Containers & Hold Times	Sediment/Soi Containers & Hold Times	Oil Containers
PCB-1016	0.1	100	1.0	2 - 1000mL glass	1 - 8 ounce glass	10mL in glass
PCB-1221	0.1	100	1.0	amber bottles	14 days HT	"
PCB-1232	0.1	100	1.0	7 days HT	"	"
PCB-1242	0.1	100	1.0	"	"	"
PCB-1248	0.1	100	1.0	"	"	"
PCB-1254	0.1	100	1.0	"	"	"
PCB-1260	0.1	100	1.0	"	"	"
PCB-1262	0.1	100	1.0	"	"	"
PCB-1268	0.1	100	1.0	"	"	"

Table 1: ENVIRONMENTAL REPORTING LIMITS (RL) FOR DEQ-DWRP LABORATORY SECTION

NPDES Scan 3 Method 608	Water Reporting Limits (ug/L)	Water Containers & Hold Times
Aldrin	0.01	2 - 1000mL glass
a-BHC	0.01	amber bottles
b-BHC	0.01	7 days HT
d-BHC	0.01	"
g-BHC (lindane)	0.01	"
BP-6 (PBB)	0.05	"
a-Chlordane	0.01	"
g-Chlordane	0.01	"
4,4'-DDD	0.05	"
4,4'-DDE	0.01	"
4,4'-DDT	0.01	"
Dieldrin	0.01	"
Endosulfan I	0.01	"
Endrin	0.01	"
Heptachlor	0.01	"
Heptachlor epoxide	0.01	"
Hexabromobenzene	0.01	"
Hexachlorobenzene	0.01	"
Hexachlorobutadiene	0.01	"
Hexachlorocyclopentadiene	0.01	"
Methoxychlor	0.05	"
Mirex	0.01	"
PCB 1016	0.1	"
PCB 1221	0.1	"
PCB 1232	0.1	"
PCB 1242	0.1	"
PCB 1248	0.1	"
PCB 1254	0.1	"
PCB 1260	0.1	"
PCB 1262	0.1	"
PCB 1268	0.1	"
Pentachlorobenzene	0.01	"
Pentachloronitrobenzene	0.01	"
Toxaphene	0.1	"
1,2,3,4-Tetrachlorobenzene	0.01	"
1,2,4,5-Tetrachlorobenzene	0.01	"

This scan is available for NPDES samples only.

Table 1: ENVIRONMENTAL REPORTING LIMITS (RL) FOR DEQ-DWRP LABORATORY SECTION

Polynuclear Aromatic Hydrocarbons (PNAs) Method 625/8270	Water Reporting Limits (ug/L)	Sediment/Soil Reporting Limits (ug/Kg)	Oil Reporting Limits (mg/Kg)	Water Containers & Hold Times	Sediment/Soil Containers & Hold Times	Oil Containers
Acenaphthene	1.0	100	100	2 - 1000mL glass	1 - 8 ounce glass	10mL in glass
Acenaphthylene	1.0	100	100	amber bottles	14 days HT	"
Anthracene	1.0	100	100	7 days HT	"	"
Benz(a)anthracene	1.0	100	100	"	"	"
Benzo(b)fluoranthene	2.0	200	200	"	"	"
Benzo(k)fluoranthene	2.0	200	200	"	"	"
Benzo(a)pyrene	2.0	200	200	"	"	"
Benzo(g,h,i)perylene	2.0	200	200	"	"	"
Chrysene	1.0	100	100	"	"	"
Dibenz(a,h)anthracene	2.0	200	200	"	"	"
Fluoranthene	1.0	100	100	"	"	"
Fluorene	1.0	100	100	"	"	"
Indeno(1,2,3-cd)pyrene	2.0	200	200	"	"	"
2-Methylnaphthalene	5.0	250	500	"	"	"
Naphthalene	1.0	100	100	"	"	"
Phenanthrene	1.0	100	100	"	"	"
Pyrene	1.0	100	100	"	"	"

PHENOLS (ACIDS) Method 625/8270	Water Reporting Limits (ug/L)	Water Containers & Hold Times
2-Chlorophenol	10	2 - 1000mL glass
4-Chloro-3-methylphenol	10	amber bottles
3/4-Methylphenol (m/p-cresol	20	7 days HT
2-Methylphenol (o-cresol	10	"
2,4-Dichlorophenol	10	"
2,4-Dimethylphenol	10	"
2,4-Dinitrophenol	50	"
2-Methyl-4,6-dinitrophenol	50	"
2-Nitrophenol	10	"
4-Nitrophenol	50	"
Pentachlorophenol	50	"
Phenol	10	"
2,4,5-Trichlorophenol	10	"
2,4,6-Trichlorophenol	10	"

Table 1: ENVIRONMENTAL REPORTING LIMITS (RL) FOR DEQ-DWRP LABORATORY SECTION

Semivolatile Organics Base/Neutral Method 625/8270	Water Reporting Limits (ug/L)	Sediment/Soil Reporting Limits (ug/Kg)	Oil Reporting Limits (mg/Kg)	Water Containers & Hold Times	Sediment/Soil Containers & Hold Times	Oil Containers
1,2,4-Trichlorobenzene	2.0	200	200	2 - 1000mL glass	1 - 8 ounce glass	10mL in glass
1,2-Dichlorobenzene	1.0	100	100	amber bottles	14 days HT	"
1,3-Dichlorobenzene	1.0	100	100	7 days HT	"	"
1,4-Dichlorobenzene	1.0	100	100	"	"	"
2,4-Dinitrotoluene	5.0	330	500	"	"	"
2,6-Dinitrotoluene	5.0	330	500	"	"	"
2-Chloronaphthalene	2.0	200	200	"	"	"
2-Methylnaphthalene	5.0	250	500	"	"	"
2-Nitroaniline	20	1,700	2000	"	"	"
3-Nitroaniline	20	1,700	2000	"	"	"
4-Bromophenyl phenylether	2.0	200	200	"	"	"
4-Chlorophenyl phenylether	1.0	100	100	"	"	"
4-Nitroaniline	20	1,700	2000	"	"	"
Acenaphthene	1.0	100	100	"	"	"
Acenaphthylene	1.0	100	100	"	"	"
Anthracene	1.0	100	100	"	"	"
Azobenzene	2.0	200	200	"	"	"
Benz(a)anthracene	1.0	100	100	"	"	"
Benzo(a)pyrene	2.0	200	200	"	"	"
Benzo(b)fluoranthene	2.0	200	200	"	"	"
Benzo(g,h,i)perylene	2.0	200	200	"	"	"
Benzo(k)fluoranthene	2.0	200	200	"	"	"
Bis(2-chloroethoxy)methane	2.0	200	200	"	"	"
Bis(2-chloroethyl)ether	1.0	100	100	"	"	"
Bis(2-chloroisopropyl)ether	1.0	100	100	"	"	"
Bis(2-ethylhexyl)phthalate	2.0	200	200	"	"	"
Butyl benzyl phthalate	1.0	100	100	"	"	"
Carbazole	10	330	1000	"	"	"
Chrysene	1.0	100	100	"	"	"
Dibenz(a,h)anthracene	2.0	200	200	"	"	"
Dibenzofuran	5.0	330	500	"	"	"
Diethyl phthalate	1.0	100	100	"	"	"
Dimethyl phthalate	2.0	200	200	"	"	"
Di-n-butyl phthalate	1.0	100	100	"	"	"
Di-n-octyl phthalate	2.0	200	200	"	"	"
Fluoranthene	1.0	100	100	"	"	"
Fluorene	1.0	100	100	"	"	"
Hexachlorobenzene	2.0	200	200	"	"	"
Hexachlorobutadiene	2.0	200	200	"	"	"
Hexachlorocyclopentadiene	10	2,000	1000	"	"	"
Hexachloroethane	1.0	100	100	"	"	"
Indeno(1,2,3-cd)pyrene	2.0	200	200	"	"	"
Isophorone	1.0	100	100	"	"	"
Naphthalene	1.0	100	100	"	"	"
Nitrobenzene	2.0	200	200	"	"	"
N-Nitrosodimethylamine	5.0	330	500	"	"	"
N-Nitrosodi-n-propylamine	2.0	200	200	"	"	"
N-Nitrosodiphenylamine	2.0	200	200	"	"	"
Phenanthrene	1.0	100	100	"	"	"
Pyrene	1.0	100	100	"	"	"

Table 1: ENVIRONMENTAL REPORTING LIMITS (RL) FOR DEQ-DWRP LABORATORY SECTION

Semivolatile Organics Base/Neutral/Acids Method 625/8270	Water Reporting Limits (ug/L)	Sediment/Soil Reporting Limits (ug/Kg)	Oil Reporting Limits (mg/Kg)	Water Containers & Hold Times	Sediment/Soil Containers & Hold Times	Oil Containers
1,2,4-Trichlorobenzene	2.0	200	200	2 - 1000mL glass	1 - 8 ounce glass	10mL in glass
1,2-Dichlorobenzene	1.0	100	100	amber bottles	14 days HT	"
1,3-Dichlorobenzene	1.0	100	100	7 days HT	"	"
1,4-Dichlorobenzene	1.0	100	100	"	"	"
2,4,5-Trichlorophenol	10	330	1000	"	"	"
2,4,6-Trichlorophenol	10	330	1000	"	"	"
2,4-Dichlorophenol	10	330	1000	"	"	"
2,4-Dimethylphenol	10	330	1000	"	"	"
2,4-Dinitrophenol	50	1,700	5000	"	"	"
2,4-Dinitrotoluene	5.0	330	500	"	"	"
2,6-Dinitrotoluene	5.0	330	500	"	"	"
2-Chloronaphthalene	2.0	200	200	"	"	"
2-Chlorophenol	10	330	1000	"	"	"
2-Methyl-4,6-dinitrophenol	50	1,700	5000	"	"	"
2-Methylnaphthalene	5.0	250	500	"	"	"
2-Methylphenol	10	330	1000	"	"	"
2-Nitroaniline	20	1,700	2000	"	"	"
2-Nitrophenol	10	330	1000	"	"	"
3/4-Methylphenol	20	660	2000	"	"	"
3-Nitroaniline	20	1,700	2000	"	"	"
4-Bromophenyl phenylether	2.0	200	200	"	"	"
4-Chloro-3-methylphenol	10	330	1000	"	"	"
4-Chlorophenyl phenylether	1.0	100	100	"	"	"
4-Nitroaniline	20	1,700	2000	"	"	"
4-Nitrophenol	50	1,700	5000	"	"	"
Acenaphthene	1.0	100	100	"	"	"
Acenaphthylene	1.0	100	100	"	"	"
Anthracene	1.0	100	100	"	"	"
Azobenzene	2.0	200	200	"	"	"
Benz(a)anthracene	1.0	100	100	"	"	"
Benzo(a)pyrene	2.0	200	200	"	"	"
Benzo(b)fluoranthene	2.0	200	200	"	"	"
Benzo(g,h,i)perylene	2.0	200	200	"	"	"
Benzo(k)fluoranthene	2.0	200	200	"	"	"
Bis(2-chloroethoxy)methane	2.0	200	200	"	"	"
Bis(2-chloroethyl)ether	1.0	100	100	"	"	"
Bis(2-chloroisopropyl)ether	1.0	100	100	"	"	"
Bis(2-ethylhexyl)phthalate	2.0	200	200	"	"	"
Butyl benzyl phthalate	1.0	100	100	"	"	"
Carbazole	10	330	1000	"	"	"
Chrysene	1.0	100	100	"	"	"
Dibenz(a,h)anthracene	2.0	200	200	"	"	"
Dibenzofuran	5.0	330	500	"	"	"
Diethyl phthalate	1.0	100	100	"	"	"
Dimethyl phthalate	2.0	200	200	"	"	"
Di-n-butyl phthalate	1.0	100	100	"	"	"
Di-n-octyl phthalate	2.0	200	200	"	"	"
Fluoranthene	1.0	100	100	"	"	"
Fluorene	1.0	100	100	"	"	"
Hexachlorobenzene	2.0	200	200	"	"	"
Hexachlorobutadiene	2.0	200	200	"	"	"
Hexachlorocyclopentadiene	10	2,000	1000	"	"	"
Hexachloroethane	1.0	100	100	"	"	"
Indeno(1,2,3-cd)pyrene	2.0	200	200	"	"	"
Isophorone	1.0	100	100	"	"	"
Naphthalene	1.0	100	100	"	"	"
Nitrobenzene	2.0	200	200	"	"	"
N-Nitrosodimethylamine	5.0	330	500	"	"	"
N-Nitrosodi-n-propylamine	2.0	200	200	"	"	"
N-Nitrosodiphenylamine	2.0	200	200	"	"	"
Pentachlorophenol	50	3,400	5000	"	"	"
Phenanthrene	1.0	100	100	"	"	"
Phenol	10	330	1000	"	"	"
Pyrene	1.0	100	100	"	"	"

Table 1: ENVIRONMENTAL REPORTING LIMITS (RL) FOR DEQ-DWRP LABORATORY SECTION

Metals	Water Reporting Limits (ug/L)	Sediment/Soil Reporting Limits (ug/Kg)	Analytical Method Reference EPA / SW-846^	MDEQ Technique	Water Containers & Hold Times	Sediment Containers & Hold Times
Aluminum	50	5,000	200.7/6010B	ICP	500 mL plastic	250 mL (8 oz.)
Aluminum	50		200.8/6020	ICP-MS	6 months HT	glass jar
Antimony	1		204.1/7040	Hydride	"	6 months HT
Antimony	1		200.8/6020	ICP-MS	"	"
Arsenic	1	500	206.3/7061A	Hydride	"	"
Arsenic	1	500	206.2/7060A	Furnace	"	"
Arsenic	1		200.8/6020	ICP-MS	"	"
Barium	5	1,000	200.7/6010B	ICP	"	"
Barium	5		200.8/6020	ICP-MS	"	"
Beryllium	1	200	200.7/6010B	ICP	"	"
Beryllium	1		200.8/6020	ICP-MS	"	"
Boron	20		200.7/6010B	ICP	"	"
Cadmium	5	2,000	200.7/6010B	ICP	"	"
Cadmium	0.2+		213.2/7131A	Furnace	"	"
Cadmium	0.2		200.8/6020	ICP-MS	"	"
Calcium	1,000	50,000	215.1/7140	FAAS	"	"
Chromium	20	2,000	200.7/6010B	ICP	"	"
Chromium	1		218.2/7191	Furnace	"	"
Chromium VI	5		7196A	DPC	24 hours HT	24 Hrs. after extraction HT
Cobalt	15	2,000	200.7/6010B	ICP	6 months HT	6 months HT
Cobalt	15		200.8/6020	ICP-MS	"	"
Copper	10	1,000	200.7/6010B	ICP	"	"
Copper	1		220.2/7211	Furnace	"	"
Copper	1		200.8/6020	ICP-MS	"	"
Iron	20	5,000	200.7/6010B	ICP	"	"
Lead	50	5,000	239.1/7420	FAAS	"	"
Lead	50	5,000	200.7/6010B	ICP	"	"
Lead	1		239.2/7421	Furnace	"	"
Lead	1		200.8/6020	ICP-MS	"	"
Lithium	8	2,000	200.7/6010B	ICP	"	"
Magnesium	1,000	50,000	242.1/7450	FAAS	"	"
Manganese	5	1,000	200.7/6010B	ICP	"	"
Manganese	5		200.8/6020	ICP-MS	"	"
Mercury	0.2	50	245.1/7470A,7471A	Cold Vapor	28 days HT	28 days HT
Mercury (LL)	.0005		245.7/1631	CVAFS	FP bottles - 28 days HT	
Molybdenum	25	5,000	200.7/6010B	ICP	500 mL plastic	6 months HT
Molybdenum	25		200.8/6020	ICP-MS	6 months HT	"
Nickel	25	5,000	200.7/6010B	ICP	"	"
Nickel	2		249.2/7521	Furnace	"	"
Nickel	2		200.8/6020	ICP-MS	"	"
Potasium	100	5,000	258.1/7610	FAES	"	"
Selenium	1	500	270.3/7741A	Hydride	"	"
Selenium	1	500	270.2/7740	Furnace	"	"
Silver	0.5+	250	272.2/7761	Furnace	"	"
Silver	0.5		200.8/6020	ICP-MS	"	"
Sodium	1,000	50,000	273.1/7770	FAAS	"	"
Strontium	5	1,000	200.7/6010B	ICP	"	"
Strontium	5		200.8/6020	ICP-MS	"	"
Titanium	10	1,000	200.7/6010B	ICP	"	"
Titanium	10		200.8/6020	ICP-MS	"	"
Thallium	2		272.2/7841	Furnace	"	"
Thallium	2		200.8/6020	ICP-MS	"	"
Vanadium	10	1,000	200.7/6010B	ICP	"	"
Vanadium	5		200.8/6020	ICP-MS	"	"
Zinc	10	5,000	200.7/6010B	ICP	"	"
Zinc	10		200.8/6020	ICP-MS	"	"

^EPA Methods for Chemical Analysis of Water and Wastes / SW-846 EPA Test Methods for Evaluating Solid Waste

FAAS = Flame Atomic Absorption Spectroscopy
FAES = Flame Atomic Emission Spectroscopy
ICP = Argon Plasma Emission Spectroscopy
DPC = Diphenylcarbazide
ICP-MS = Inductively Coupled Plasma - Mass Spectrometry
CVAFS = Cold Vapor Atomic Fluorescence Spectrometry

* "Standard Methods For the Examination of Water and Wastewater"
+ = Matrix Dependent
FP = Fluoropolymer Bottles

Revision: 12/2002

Table 1: ENVIRONMENTAL REPORTING LIMITS (RL) FOR DEQ-DWRP LABORATORY SECTION

Non-Metals	Water Reporting Limits (ug/L)	Analytica Method Reference EPA / SW-846^	MDEQ Technique	Sampling Containers (waters)	Hold Times
Alkalinity	20,000	310.1	Auto Analyzer	500 mL plastic	14 days
Alkalinity, Bicarbonate	10,000		Calculate	"	"
Alkalinity, Carbonate	10,000		Calculate	"	"
Ammonia	10	350.1	Auto Phenolate	"	28 days
BOD-Carb.	2000	405.1	5 Day-DO Probe	"	48 hours
BOD-Total	2000	405.1	5 Day-DO Probe	"	"
Chloride	1000	325	Auto Analyzer	"	28 days
COD (high Level)	100,000	410.4	Colorimetric	"	"
COD (low level)	5000	410.4	Colorimetric	"	"
Conductivity	1.0*	120.1	Bridge	"	"
Cyanide	5	335.2/9010	Man. Dist., Man. PBA Color	"	14 days
Dissolved Oxyger	100	360.2	Manual Titration	250 mL glass	8 hours
Hardness (Ca2CO3)	5000	130.2	Calculate	500 mL plastic	6 months
Nitrate + Nitrite	10	353.2	Auto Cd Reduction	"	28 days
Nitrite	10	353.2	Auto Diazotization	"	48 hours
Nitrogen, Kjeldah	100	351.2	BD, Auto Salicylate	"	28 days
OrthoP	10	365	Auto Ascorbic Acid Reduction	"	48 hours
Phenolics	10	420.2	Auto Dist., Auto 4AAP	500 mL glass	28 days
Phenolics	10	420.2/9066	Manual Dist., Auto 4AAF	"	"
Phosphorous, Tota	10	365.4	BD Auto Ascorbic Acid Reduc.	500 mL plastic	28 days
Residue	20,000	160.1	Total Filt-TDS 180C	"	7 days
Residue	4000	160.2	Non Filt-Susp. Sol. 105C	"	"
Silicates	50	370.1	Auto Ascorbic Acid Reduction	"	28 days
Sulfate	2000	375.1	Auto Analyzer	"	"
Sulfide	20	376.2	Methylene blue	250 mL plastic	7 days
TOC	500	415.2	UV/Persulfate (DC-80)	500 mL plastic	28 days
Turbidity	0.4#	180.1	Turbidimeter	"	48 hours
Oil & Grease	10,000	1664	SPE	2-250 mL glass	28 days

^EPA Methods for Chemical Analysis of Water and Wastes / SW-846 EPA Test Methods for Evaluating Solid Was

* = umhos/cm
\# = NTU

Calculate = Value is calculated from existing dat
Bridge = Conductivity mete
DO Probe = Dissolved Oxygen (YSI) Prob
Cd Reduc. = Cadmium Reduction
BD = Block Digester
Man. Dist.= Manual Distillation
SPE = Solid Phase Extraction

4AAP = 4 Amino Antipyrene
BOD = Biochemical Oxygen Demand
COD = Chemical Oxygen Demand
TOC = Total Organic Carbon
TDS = Total Dissolved Solids
Auto Dist.= Automated Distillation

Table 1: ENVIRONMENTAL REPORTING LIMITS(RL) FOR DEQ-DWRP LABORATORY SECTION

VOLATILE ORGANICS Method TO-15	Air Reporting Limits (ppbv)	Air Reporting Limits (ug/M3)	Air Containers
Dichlorodifluoromethane	0.3	1.5	6L Canister
Chloromethane	0.3	0.6	"
1,2-Dichloro-1,1,2,2-tetrafluoroethane	0.3	2.1	"
1,3-Butadiene	0.3	0.7	"
Vinyl Chloride	0.3	0.8	"
Bromomethane	0.3	1.1	"
Chloroethane	0.3	0.8	"
Acetonitrile	1.0	1.7	"
Trichlorofluoromethane	0.3	1.7	"
Acrylonitrile	0.5	1.1	"
1,1-Dichloroethylene	0.3	1.2	"
Methylene Chloride	0.3	1.0	"
1,1,2-Trichloro-1,2,2-Trifluoroethane	0.3	2.3	"
Trans-1,2-Dichloroethylene	0.3	1.2	"
1,1-Dichloroethane	0.3	1.2	"
Methyl Tert-Butyl Ether	0.5	1.8	"
Methyl Ethyl Ketone	5.0	14.5	"
2-Chloro-1,3-Butadiene	0.3	1.1	"
Cis-1,2-Dichloroethylene	0.3	1.2	"
Hexane	1.0	3.5	"
Chloroform	0.3	1.4	"
1,2-Dichloroethane	0.3	1.2	"
1,1,1-Trichloroethane`	0.3	1.6	"
Benzene	0.3	0.9	"
Carbontetrachloride	0.3	1.9	"
1,2-Dichloropropane	0.3	1.4	"
Bromodichloromethane	0.3	2.0	"
Trichloroethylene	0.3	1.6	"
2,2,4-Trimethylpentane	0.3	1.4	"
Cis-1,3-Dichloropropylene	0.3	1.3	"
Methyl Isobutyl Ketone	1.0	4.0	"
Trans-1,3-Dichloropropylene	0.3	1.3	"
1,1,2-Trichloroethane	0.3	1.6	"
Toluene	0.3	1.1	"
Dibromochloromethane	0.3	2.5	"
1,2-Dibromoethane	0.3	2.3	"
Tetrachloroethylene	0.3	2.0	"
Chlorobenzene	0.3	1.4	"
Ethylbenzene	0.3	1.3	"
m&p-Xylene	0.3	1.3	"
Bromoform	0.3	3.0	"
Styrene	0.3	1.3	"
1,1,2,2-Tetrachloroethane	0.3	2.0	"
o-Xylene	0.3	1.3	"
1,3,5-Trimethylbenzene	0.3	1.4	"
1,2,4-Trimethylbenzene	0.3	1.4	"
Benzyl Chloride	0.3	1.5	"
1,3-Dichlorobenzene	0.3	1.8	"
1,4-Dichlorobenzene	0.3	1.8	"
1,2-Dichlorobenzene	0.3	1.8	"
1,2,4-Trichlorobenzene	0.3	2.2	"
Hexachloro-1,3-Butadiene	0.3	3.1	"

Table 1: ENVIRONMENTAL REPORTING LIMITS (RL) FOR DEQ-DWRP LABORATORY SECTION

Aldehydes Method TO-11	Air Reporting Limits (ug)	Containers Hold Time
Formaldehyde	0.30	DNPH Cartridges
Acetaldehyde	0.30	14 Days for Extraction
Acetone	0.30	"
Propionaldehyde	0.30	"
Crotonaldehyde	0.30	"
n-Butyraldehyde	0.30	"
Benzaldehyde	0.40	"
Isovaleraldehyde	0.40	"
Valeraldehyde	0.40	"
o-Tolualdehyde	0.40	"
m,p-Tolualdehyde	0.40	"
Hexanaldehyde	0.40	"
2,5-Dimethylbenzaldehyde	0.40	"

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

LABORATORY SERVICES SECTION
ENVIRONMENTAL LABORATORY
QUALITY ASSURANCE PROGRAM PLAN

Figure 2-1

Organization Chart

LABORATORY SERVICES SECTION

March 3, 2003

D SCOTT
Secretary 9
SECRTRYAG45R

LABORATORY SERVICES SECTION
B AVERY
Lab Scientist Mgr 15
LABSMGR4A02N

L FELTON
Student Assistant
STUDASTEF25N

DRINKING WATER UNIT
S KERNS
Lab Scientist Mgr 14
LABSMGR3A13N

- **C DANIELS** — Lab Sci P11 — LABSCIEA13R
- **P CRAWFORD** — Lab Tech 11 — LABYTCHAA08R
- **R EATON** — Lab Tech E10 — LABYTCHEA32R
- **P KORYCIAK** — Lab Tech E10 — LABYTCHEA32R
- **J McLEOD** — Lab Tech E10 — LABYTCHEA32R
- **S NWABUDE** — Lab Tech 11 — LABYTCHAA08R
- **K MROCZKOWSKI** — Lab Tech 8 — LABYTCHEA32R
- **S. REYNOLDS** — Lab Scientist 10 — LABSCIEA13R
- **M BRISENO** — Lab Scientist P11 — LABSCIEA13R
- **J GUYER** — Lab Scientist P11 — LABSCIEA13R
- **K JACKSON** — Lab Scientist P11 — LABSCIEA13R
- **PK NAIR** — Lab Scientist P11 — LABSCIEA13R
- **J PIEPER** — Lab Scientist 12 — LABSCIAA11R
- **VACANT** — Lab Scientist P11 — LABSCIEA13R
- **W. COON** — Lab Scientist P11 — LABSCIEA13R
- **VACANT** — Student — STUDASTE

ENVIRONMENTAL INORGANIC UNIT
S GREGG
Lab Scientist Mgr 14
LABSMGR3A12N

- **VACANT** — SA — STUDASTEF27N
- **VACANT** — Lab Scientist 12 — LABSCIAA12R
- **L UTESS** — Med Tech Spv 12 — MEDTSPV1A05N
- **B BAKER** — Lab Tech E10 — LABYTCHEA34R
- **T KUHN** — Lab Tech 8 — LABYTCHEA10R
- **B GRENIER** — Lab Tech 11 — LABYTCHAA10R
- **VACANT** — Lab Tech E10 — LABYTCHEA34R
- **G WILLIAMS** — Lab Tech 11 — LABYTCHAA10R
- **L. VIETZKE** — Lab Scientist 12 — LABSCIAA12R
- **R ANDERSON** — Lab Scientist 12 — LABSCIAA12R
- **M BERTHOLD** — Lab Scientist 10 — LABSCIEA14R
- **D BRUDNYAK** — Lab Scientist P11 — LABSCIEA14R
- **E GILLIAM** — Lab Scientist P11 — LABSCIEA14R
- **M JERSIN** — Lab Scientist P11 — LABSCIEA14R
- **B NELSON** — Lab Scientist P11 — LABSCIEA14R
- **K SHANE** — Lab Scientist P11 — LABSCIEA14R
- **D SLABBEKOORN** — Lab Scientist P11 — LABSCIEA14R
- **T SONNENBERG** — Lab Scientist P11 — LABSCIEA14R

ENVIRONMENTAL ORGANIC UNIT
C SMITH
Lab Scientist Mgr 14
LABSMGR3A14N

- **T MACALINCAG** — SEEP
- **N HUDE** — SA — STUDASTEF28N
- **VACANT** — SA — STUDASTEF28N
- **S IRRER** — Main Mech E9 — MAINMCHEB28R
- **E GAFNER** — Equip Tech 10 — EQIPTCHEA08R
- **R LEE** — Equip Tech E10 — EQIPTCHEA08R
- **M LOTOSZINSKI** — Equip Tech 11 — EQIPTCHAA16R
- **K DeVRIES** — Lab Tech E10 — LABYTCHEA33R
- **VACANT** — Student — STUDASTEF28N
- **J BRONSON** — Lab Scientist P11 — LABSCIEA24R
- **F CALERA** — Lab Scientist Spl 13 — LABSCIAA12R
- **M FERRIS** — Lab Scientist P11 — LABSCIEA15R
- **S HAYWARD** — Lab Scientist P11 — LABSCIEA23R
- **K LI** — Lab Scientist P11 — LABSCIEA21R
- **P RUMLER** — Lab Scientist 12 — LABSCIAA13R
- **JR SCOTT** — Lab Scientist P11 — LABSCIEA25R
- **S TAIT** — Lab Scientist 12 — LABSCIAA13R
- **VACANT** — Lab Scientist 9-P11 — LABSCIEA22R

SYSTEMS MANAGEMENT UNIT
G KRISZTIAN
Lab Scientist Mgr 14
LABSMGR3A15N

- **D COON** — Sec E8 (PI) — SECRTRYEH66R
- **K BINNEY** — Dept Tech 10 — DPTLTCHAB49R
- **B FELDPAUSCH** — SA — STUDASTEF27N
- **S BREWSTER** — Data Coding Opr E7 — DATAOPREB37R
- **S HEMPSTED** — Data Coding Opr E7 — DATAOPREB37R
- **S RATHBURN** — Data Coding Opr E7 — DATAOPREB37R
- **D WHITE** — Storekeeper E6 — STORKPREA96R
- **D JOHNSON** — Comm Asst E7 — COMNASTEB92R
- **A LEE** — Lab Scientist 12 — LABSCIAA14R
- **C PRICE** — Lab Asst E8 — LABYASTEA07R
- **R DULIBAN** — Lab Tech 8 — LABYTCHEA35R
- **VACANT** — Student — STUDASTEF27N

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LAB SERVICES SECTION

EFFECTIVE DATE: 02/2007 SOP# 400 REVISION # 2

GEL-PERMEATION CHROMATOGRAPHY (GPC)

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1.0 Applicable Analytical Methods

1.1 Gel-Permeation Cleanup, U.S. Environmental Protection Agency, Method 3640C, SW846, Revision 1, September 1994.

1.2 Quality Control, U.S. Environmental Protection Agency, Chapter 1, SW846, Revision 1, July 1992.

1.3 Organic Extraction and Sample Preparation, U.S. Environmental Protection Agency, Method 3500B, SW846, Revision 2, December 1996.

1.4 Cleanup, U.S. Environmental Protection Agency, Method 3600C, SW846, Revision 3, December 1996.

2.0 Matrix or Matrices

2.1 Aqueous.

2.2 Soil.

2.3 Sediment.

2.4 Sludge.

2.5 Clay.

2.6 Oil.

2.7 Solid or liquid waste.

3.0 Method Detection Limits (MDLs)

3.1 All MDLs shall be performed according to the Code of Federal Regulations 40 CFR, Part 136, Appendix B. See internal procedure SOP 103.

3.2 MDLs are extracted according to internal procedures SOP 401 or SOP 405.

4.0 Scope and Application

4.1 GPC is a size exclusion cleanup. The packing gel is porous. The size of the packing material is such that it excludes molecules larger than the compounds of interest. The compounds of interest can be found in internal procedures SOP 500 and SOP 502.

4.2 GPC can be used for both polar and non-polar analytes.

4.3 GPC is most effective at removing high boiling materials that condense in the injection port of a gas chromatograph (GC) or in the front of a GC column. This residue usually reduces separation efficiency because of adsorption of the target analytes on the active sites. Pentachlorophenol is especially susceptible to this problem.

5.0 Method Summary

5.1 Water samples are extracted by following internal procedure SOP 405. Soil samples are extracted by following internal procedure SOP 401. Oil samples are prepared as defined in internal procedure SOP 404. All extracts must be in methylene chloride.

5.2 The column is purchased prepackaged with Envirosep-ABC bio-beads or Envirogel. The column is continuously flushed with methylene chloride. The column is calibrated. Sample extract is loaded onto the column. A fraction of the eluate is collected and then concentrated.

6.0 Definitions

6.1 Method Preparation Blank (MPB) – An aliquot of a blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and surrogates that are used with samples.

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6.2 Laboratory Control Sample (LCS) – An aliquot of blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is treated exactly as a sample including extraction, cleanup, and analysis.

6.3 Matrix Spike (MS) – An aliquot of a sample to which known quantities of the target analytes are added in the laboratory. The MS is treated exactly as a sample including extraction, cleanup, and analysis. The background concentrations of the analytes in the sample must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

6.4 Matrix Spike Duplicate (MSD) – A second aliquot of sample to which known quantities of the target analytes are added in the laboratory. The MSD is treated exactly as a sample including extraction, cleanup, and analysis. The background concentrations of the analytes in the sample must be determined in a separate aliquot and the measured values in the MSD corrected for background concentrations. Analyses of the MS and MSD indicate precision associated with laboratory procedures.

7.0 Interferences

7.1 Interferences from contaminants can occur from solvents, glassware, and other sample processing equipment. Pesticide quality solvents (or better) and solvent rinsing of all glassware should help minimize contaminants.

7.2 The use of plastic must be minimized to prevent phthalate contamination.

7.3 Soap residue must be thoroughly rinsed from all glassware to avoid degradation of some pesticide compounds.

7.4 Interferences from compounds with similar molecular size to the target analytes will not be removed by this method.

8.0 Safety

8.1 Safety glasses are required in all designated laboratory areas.

8.2 Be familiar with the Laboratory Chemical Hygiene Plan.

8.3 Be familiar with the Laboratory Safety Policy (internal procedure SOP 100).

8.4 Hexane, 1-chlorobutane, acetone, methyl alcohol, and methylene chloride material safety data sheets (MSDSs) are attached.

8.5 Nitrile or other solvent resistant gloves must be worn when handling solvents.

9.0 Equipment and Supplies

9.1 Culture tubes – Glass, 16 mm x 100 mm, VWR catalog #60825-618 or equivalent.

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9.1.1 Place 2 or 10 mL of hexane into each tube from a calibrated bottle top dispenser. Check the dispenser with a volumetric flask prior to use.
9.1.2 Mark the meniscus with a lab marker.
9.1.3 Dispose of the hexane. Let tube air dry.
9.1.4 Hexane may also be saved for reuse. Transfer used hexane to the container labeled "for pre-marking only."

9.2 Snap caps – Zymark, "star" cut, part #ZB7216.

9.3 Aluminum foil.

9.4 Vials – 2 mL crimp cap, HP #5181-3375.

9.4.1 Place 1 mL of hexane into each vial from a calibrated bottle top dispenser. (Section 9.9) Check the dispenser with a volumetric flask prior to use.
9.4.2 Mark the meniscus with a lab marker.
9.4.3 Dispose of the hexane. Let vial air dry.
9.4.4 Hexane may also be saved for reuse. Transfer used hexane to the container labeled "for pre-marking only."

9.5 Crimp caps – HP #5181-1210.

9.6 Turbovap tubes – 50 and 250 mL capacity.

9.7 Turbovap concentration workstation – With temperature control up to 95°C and nitrogen pressure from zero to 20 psi (usual settings – temperature = 35°C and pressure = 0 – 15 psi).

9.8 Pasteur pipets – Disposable, glass.

9.9 Bottle top solvent dispensers – adjustable.

9.9.1 Calibrate to desired volume by filling appropriate volumetric flasks and adjusting dispenser.

9.10 Pipets – Glass, class A, various sizes.

9.11 Volumetric flasks – Various sizes.

9.12 Column – Envirosep-ABC, 350 x 21.2 mm, Phenomenex #OOW-3035-PO, or Waters Envirogel GPC, 300 x 19mm, Waters #WAT036554 and Waters Envirogel GPC 150 x 19MM, Waters #WAT036555.

9.13 Guard column – Envirosep-ABC, 60 x 21.2 mm, Phenomenex #03R-3035-PO, or Waters Envirogel GPC, 30 x 4.6mm, Waters #186001913.

9.14 UV detector.

9.14.1 J2 Scientific DT0001, or equivalent.
9.14.2 ISCO UA-6, or equivalent.

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9.14.2.1 Noise filter – 1.5 sec.
9.14.2.2 Peak separator – 0.3 min.
9.14.2.3 Chart speed
9.14.2.3.1 Calibration – 30 cm/hr.
9.14.2.3.2 Samples – 3 cm/hr.

9.14.2.4 Sensitivity – 0.1 abs

9.15 Zymark (Caliper) benchmate preparation station, OI GPC 2000, J2 AccuPrep GPC System, or equivalent.

9.16 ISCO Foxy 200 fractionator/collector – For use with the Zymark (Caliper) benchmate preparation station.

9.17 SSI Model 300 LC pump or equivalent – For use with the Zymark (Caliper) benchmate preparation station.

9.17.1 Speed

9.17.1.1 Calibration and analysis – 5 setting on pumps. The measured flow should be between 4.5 and 5.5 mL/minute. Adjust setting as necessary.
9.17.1.2 Standby mode – 0.2.

9.18 Computer –

9.18.1 With Zymark (Caliper) benchmate software.
9.18.2 With OI Analytical software.
9.18.3 With J2 Scientific software.

9.19 Bottles – Amber, with teflon lined caps.

9.20 Filters – Acrodisc, 0.45 µm membrane syringe filter, Pall Life Science #4219, VWR #28143-924.

9.21 Syringes – 10 mL, slip tip, disposable, plastic.

9.21.1 Slip tip, disposable, plastic (for pesticide/PCBs).
9.21.2 Luer lock, glass, Hamilton #1010 or equivalent (for semi-volatiles).

9.22 Vortex mixer.

9.23 Tape.
9.24 Tubes – Screw cap, glass, 13 x 100 mm, VWR #60826-701.

9.24.1 Place 2 or 5 mL of hexane into each tube from a calibrated bottle top dispenser. (Section 9.9)
9.24.2 Mark the meniscus with a lab marker.
9.24.3 Dispose of the hexane. Let the tube air dry.

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9.24.4 Hexane may also be saved for reuse. Transfer used hexane to the container labeled "for pre-marking only."

9.25 Screw caps – For tubes, VWR #60827-110.

9.26 Teflon liners – For screw caps, Thomas Scientific #2390J60.

10.0 Reagents and Standards

10.1 All reagents, solvents, and standards must be traceable to the stock inventory tracking log. (See Attachment 22.1)

10.2 All reagents, solvents, and standards must be labeled with: date received, date opened, expiration date, tracking number, and receiver's initials.

10.3 All prepared reagents and standards must be labeled with: date prepared, expiration date, preparer's initials, tracking number, diluent, and description.

10.4 All standard logbooks must be completely filled out.

10.5 Nitrogen – Liquid.

10.6 Hexane (95% n-hexane) – Pesticide grade, demonstrated to be free of target analytes.

10.7 Methylene chloride (MeCl)– Pesticide grade, demonstrated to be free of target analytes.

10.8 GPC calibration solution – Absolute Standards, part #20041, in methylene chloride, 5 mL.

Corn oil	250 mg/mL
Bis (2-ethylhexyl phthalate)	10 mg/mL
Methoxychlor	2 mg/mL
Perylene	0.2 mg/mL
Sulfur	0.8 mg/mL

10.8.1 Place approximately 60 mL of methylene chloride (Section 10.7) into a 100 mL volumetric flask.
10.8.2 Open two ampules of GPC calibration solution. Pipet a total of 10 mL into the volumetric flask.
10.8.3 Dilute to the mark with methylene chloride. Mix.
10.8.4 Transfer to an amber bottle containing a teflon lined cap. Label.
10.8.5 Fill out the semi-volatile standard logbook (Attachment 22.2). Standard is good for up to six months.
10.8.6 Store in a refrigerator.

10.9 1-chlorobutane – J.T. Baker, E738-07 or equivalent.

10.9.1 Zymark (Caliper) – 2 mL run as samples.
10.9.2 J2 and OI – 2 mL and 8 mL MeCl, two filtered and run as samples.

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10.10 Helium gas.

10.11 Acetone – HPLC grade, demonstrated to be free of target analytes.

10.12 Methyl Alcohol – HPLC grade, demonstrated to be free of target analytes.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Aqueous samples – See internal procedure SOP 405.

11.2 Solid samples – See internal procedure SOP 401.

11.3 Waste samples – See internal procedure SOP 404.

11.4 Holding times – Sample extracts may be analyzed for up to 40 days from the extraction date.

11.5 Storage – Sample extracts must be stored in a refrigerator until GPC cleanup. After this, samples are stored in crimp cap vials, at 4ºC or below, and protected from light until analysis.

11.5.1 Pesticide/PCB soil and oil samples also have an aliquot stored in 13 X 100 mm vials (Section 9.24) for possible additional cleanup or analysis.

12.0 Quality Control (QC)

12.1 All associated QC samples must be processed through this cleanup method along with the sample extracts.

12.2 Extracted sample batches.

12.2.1 Extracted sample batches consist of a maximum of 20 samples, 1 MPB, 1 MS, 1 MSD, 1 LCS, and 1 LCSD.

12.2.2 MSs and MSDs are actual samples that are selected at random from within the batch of 20.

12.2.3 All 20 samples and the QC samples are spiked with known surrogate concentrations. The recoveries for these compounds are used as indicators of possible problems that may occur during extraction or during cleanup of individual samples.

12.2.4 The MS, MSD, LCS, and LCSD are spiked at a known concentration with target analyte compounds. The replicate MSs or LCSs are used to evaluate the entire batch for precision and accuracy.

12.3 Initial demonstration of ability (IDOA) – Each analyst must demonstrate initial proficiency with this method by generating data of acceptable accuracy and precision for target analytes. This is accomplished by extraction and cleanup of four aliquots of a reference sample.

12.3.1 Five aliquots of reagent water or sand are extracted using internal procedures SOP 401 or SOP 405. One of these is a MPB. The other four are spiked at a mid-range concentration with all of the analytes of interest.

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12.3.2 Sample extracts are taken through all the normal sample cleanup methods.
12.3.3 Extracts are then analyzed by GC-electron capture detector or gas chromatograph/mass spectrometry. See internal procedures SOP 500 or SOP 502.

13.0 Calibration and Standardization

13.1 All GPC columns must be calibrated at least once per week unless not in use.

13.2 All GPC calibration information must be completed in a calibration logbook. Room temperature must also be recorded.

13.3 Zymark (Caliper) GPC.

NOTE: *The Zymark (Caliper) benchmates are never turned off unless there is a problem.*

13.3.1 Make sure that the pump is on. Check to make sure that there is sufficient MeCl in the reservoir. Fill if needed. Adjust the flow to 5.0. Mobile phase is continuously sparged with helium.
13.3.2 Empty waste container into steel drum labeled "Chlorinated Waste."
13.3.3 Turn the detector on (Section 9.14). The green "ready" light will indicate when the detector is warmed up. Make sure that the pen is inserted into the recorder and that the cap is off. Check to make sure that the sensitivity is set at 0.1. Check to make sure that the chart speed is set at 30 cm/hr.
13.3.4 Calibration solution preparation.

- **13.3.4.1** Pipet 1 mL of GPC calibration solution (Section 10.8.4) into each of two culture tubes (Section 9.1).
- **13.3.4.2** Place the tube in position 1, rack 1, on the benchmate #16, and in the appropriate position for the program, rack 1, on the benchmate #14 and #15.
- **13.3.4.3** Place waste containers in positions 1 and 2 on the Foxy 200 of all benchmates.
- **13.3.4.4** Place a tube with 1 mL of MeCl in position 2, rack 1, for the benchmate #16, and in the appropriate position for the program, rack 1, for the benchmate #14 and #15.
- **13.3.4.5** Minimum of 4 rinses in between calibration solution and samples. Either after calibration run or at beginning of sample batch.

13.3.5 Turn the Foxy 200 on by choosing "run." Leave it in the "waiting for external (re)start" position.
13.3.6 Check the filter holder. Fill as needed. Place the writing side of the filter in the "up" position.
13.3.7 Zero the recorder by choosing "autobaseline." Adjust the recorder offset to the first heavy line on the right side of the recorder paper.
13.3.8 Place the appropriate benchmate calibration diskette into the benchmate. Choose "load." (See Attachment 22.3 for the programs).

NOTE: *The diskettes for the benchmate I (#14 and #15) are not compatible with the benchmate II (#16) and vice versa.*

13.3.9 When the runs are completed, choose "stop" on the Foxy 200. Return the pump to 0.2 mL/min. if no samples are to be run. Return the chart speed to 3. Turn the detector off.

13.3.10 Remove the diskette from the benchmate when the run is completed (as signified by a beep or when the run light is off).

13.3.11 Proceed to section 15 for calculations.

13.4 OI Analytical GPC

13.4.1 Check to make sure that there is sufficient MeCl in the reservoir. If necessary, add more MeCl. Sparge with helium.

13.4.2 Empty waste container into the steel drum labeled "chlorinated waste."

13.4.3 Turn the detector on (Section 9.14.2). The green "ready" light will indicate when the detector is warmed up. Make sure that the pen is inserted into the recorder and that the cap is off. Check to make sure that the sensitivity is set at 0.1. Check to make sure that the chart speed is set at 30 cm/hr.

13.4.4 Choose the "WinSEP" icon on the computer. (See Attachment 22.18 for the program.) Choose "on/off" located in the front of the GPC. The "run" light should come on. Run in "bypass" mode until priming is completed.

13.4.5 Choose "prime" on the front of the GPC. Attach a luerlock syringe to the prime port on the right side of the unit. Open the black knob by turning counterclockwise. Slowly draw the air out of the GPC lines by pulling back on the syringe plunger. Close the black knob. Repeat as needed until all bubbles are removed from the system.

13.4.6 Choose "prime" again. In the "WinSEP" program, open the "status" window. Choose "prime syringe." Check the coiled tubing on the right side of the GPC for bubbles. Choose "prime syringe" again if bubbles are observed.

13.4.7 Open the "status" window. Choose "calibration" from the "sequence name" pull down menu. Check to make sure that it is correct.

13.4.8 Select "equilibrate" in the "status" window. The run will automatically switch from "bypass" mode to "in line." Equilibration takes approximately 30 minutes. The pressure should be approximately 250 psig. This can be checked in the "status" window. Choose "autobaseline" on the UV detector. Adjust the recorder pen to the first heavy line from the right edge using the "recorder offset" knob.

13.4.9 Calibration solution preparation – Take out of refrigerator to warm up (Section 10.8).

13.4.9.1 Pipet 1 mL of GPC calibration solution (Section 10.8.4) into a 10 mL pre-marked culture tube (Section 9.1).

13.4.9.2 Dilute to the mark with methylene chloride.

13.4.9.3 Attach an acrodisc filter (Section 9.20) to a syringe (Section 9.21).

13.4.9.4 Pour diluted calibration solution into the syringe. Filter into a culture tube (Section 9.1). The tube does not need foil.

13.4.9.5 Place the tube in the second spot in the sample tray.

13.4.10 Place a rinse tube of 10 mL filtered MeCl into the first and third spots in the sample tray.

13.4.11 When equilibration has finished and when the baseline is free of interferences, choose "start" in the "status" window. When the instrument begins the "dump" stage of the calibration sample, make a mark on the chromatogram by adjusting the "recorder offset" knob on the front of the recorder.

13.4.12 When the run is completed, the pump will turn off automatically. Recap the chart recorder pen when not in use.

13.4.13 Proceed to section 15 for calculations.

13.5 J2 Scientific GPC

13.5.1 Check to make sure there is sufficient MeCl in the reservoir. If necessary, add more MeCl.

13.5.2 Turn detector on. The green "ready" light will indicate when the detector is warmed up.

13.5.3 Choose the Accuprep icon on the computer. When program opens, choose "Use Autoinjector." Allow unit to initialize.

13.5.4 Click on the "Solvent Pump" icon. Click "Enable." Click "OK."

13.5.5 Prime solvent pump by removing top screw and screwing in priming syringe. Withdraw air by slowly pulling back on syringe plunger. When MeCl flows freely, remove syringe and replace top screw.

13.5.6 In AccuPrep program, choose "Prime Syringe." Observe syringe pump. If syringe pump pulls out air, prime again.

13.5.7 On main screen of AccuPrep, click "Put In Line" to put the column in line. Autozero the sensor on the main screen. The pressure should be around 250psig.

13.5.8 Open the Sequence shortcut by clicking on the sequence icon. In "Designation (ID)" #1, type "Rinse #1." In ID #2, type "#CalMMDDYY," where "#" is the unit number (1 or 2), and MMDDYY is the date of the calibration, i.e., December 2, 2004, would be 120204. In ID #3, type "Rinse#3." Change sample type to reflect what the sample is. Calibration solutions are "Calibration," rinse solutions are "Solvent Blank."

13.5.9 Save sequence as "#calMMDDYY," using the name format as step 13.5.8.

13.5.10 Prepare Calibration solution as in 13.4.9.

13.5.11 Place a rinse tube of 10 mL filtered Methylene Chloride in first and third spot of sample tray.

13.5.12 Run sequence.

13.5.13 After sequence is finished, click the AccuChrome button. When AccuChrome opens, click File, Import V 1.0 Data. Ensure that browser opens in correct unit data – J2 #63 or J2 #64. If in the wrong browser, search back until in the correct J2 system, then click on folder "Data." Files should end in "XML," NOT ".seq."

13.5.14 Double click on file that was saved when sequence was made. Depending on number of samples, it may take a few minutes to open. Left side of screen will be blank when file is loaded. Click on cylinder in upper left corner. Search through dates and times for correct runtime of sequence.

13.5.15 Click on date and time. Double lick on "Sequence #calXXXX." Double click "Sample [2] #calXXXX" Double click "Method." Double click "Data Plot."

13.5.16 When plot opens, click "Find Peaks." Adjust baseline by clicking, holding and moving the purple diamond. Adjust peak lines by clicking and holding triangle associated with line. When all lines are correct, click "Store."

13.5.17 On top tool bar, click "Reports," then "Column Calibration Report." Click Print. Computer automatically generates retentions and if results pass or fail. (See Attachment 22.22)

13.5.18 Proceed to Section 15 for calculations.

14.0 Procedure

14.1 Zymark (Caliper) GPCs.

14.1.1 Make sure the pump is on. Check to make sure that there is sufficient MeCl in the reservoir. Fill and sparge if needed. Adjust the flow to 5.0.

14.1.2 Empty all waste containers into the waste drum labeled "chlorinated waste."

14.1.3 Check the filter holder. Fill as needed.

14.1.4 Turn the detector on (Section 9.14.2). The green "ready" light indicates when the detector is warmed up. Make sure that the pen is inserted into the recorder and that the cap is off. Check to make sure that the sensitivity is set at 0.1. Make sure that the chart speed is 3 cm/hr. Adjust the baseline by choosing "recorder offset" or "auto baseline."

14.1.5 Turn the Foxy 200 sample collector on. Adjust the window if necessary (section 15). Leave it in the "waiting for external signal" position. (See Attachment 22.6 for the program if the power has been out or if the window needs to be adjusted).

14.1.6 Sequence creation.

14.1.6.1 Place the appropriate program disk into the computer. The programs are listed in Attachment 22.4 for each benchmate.

NOTE: *The diskettes for the benchmate I (#14 and #15) are not compatible with the benchmate II (#16) and vice versa.*

14.1.6.2 Choose the icon for the correct Benchmate.

14.1.6.3 Choose "sample data." Choose "input."

14.1.6.3.1 Heading 1 contains the name of the program.

14.1.6.3.2 Heading 2 contains the batch number(s) and the type of scan.

14.1.6.3.3 Heading 3 contains the analyst's initials.

14.1.6.3.4 The initial volume is 2 or 10.

14.1.6.4 Determine which program to use and its order on the diskette prior to entering sample numbers into the spreadsheet. The benchmate reads program #1 first, program #2 second, etc. The sample number information should be entered starting on the same line that corresponds to the order in which the program is on the diskette (i.e., if the program required is #3 on the diskette, the first sample of the run should be entered on the third line, and the first sample should go into position #3 on the benchmate).

14.1.6.5 Enter the sample number. Choose "enter." Choose the "enter" again. Continue entering up to 18 samples in this manner (36 for the dual injector). Choose "*I*." Choose "P" (Attachment 22.5).

14.1.6.6 Tape the printout to the hood above the benchmate. Choose "*I*." Choose "save." Choose "done." Choose "exit" two times. Take the disk out.

14.1.7 Loading samples

14.1.7.1 Soil samples are extracted according to internal procedure SOP 401. Oils are diluted according to internal procedure SOP 404. Two mL of extract is placed into a culture tube (Section 9.1). Samples may be diluted to 10 mL if GPC is programmed for a 10 mL start volume. Samples that are unusually thick should be pre-filtered (Attachment 22.16). All extracts must be in MeCl. Foil is placed over the tube and held in place with a star cap (Section 9.2).

14.1.7.2 Label each sample tube using a marker and a labeled piece of tape.

14.1.7.3 Single injector loading – Load samples into rack 1 of the benchmate according to the printout (#1 is the bottom right spot, and #11 is directly behind it).

NOTE: *The Benchmate moves from right to left and from front to back.*

14.1.7.4 Dual injector loading – Samples should be loaded alternately onto rack 1. The benchmate #14 uses the odd-numbered spots. The benchmate #15 uses the even-numbered spots.

14.1.7.5 The Foxy 200 requires samples to be in increments of 3. Place MeCl rinses in any remaining spots. Both batches must contain the same number of tubes in rack 1 and must also be divisible by three for the dual injector.

14.1.8 Load rack 3 with transfer tubes (Section 9.1). Load enough empty tubes to match the number and placement in rack 1.

14.1.9 If pesticides/PCBs are to be analyzed, place 30 mL of hexane into rinsed 250 mL turbovap tubes. If semi-volatiles samples are to be analyzed, use empty turbovap tubes. Place them on the Foxy 200. Transfer the sample ID tape from the sample tubes to the turbovap tubes. The Foxy 200 moves from left to right and from front to back (#1 is the bottom left spot, and #4 is directly behind it).

14.1.10 Foxy 200 operation – Refer to Attachment 22.6. Settings should rarely be changed, with the exception of window settings.

14.1.11 Choose “Run A or B” on the Foxy 200 to return the autosampler arm to the “wait” position. The screen should read “waiting for external signal.”

14.1.12 Place the benchmate diskette (Section 14.1.6) into the benchmate. Choose “load.”

14.1.13 The Foxy 200 shuts the pump off when the run is completed. Turn the pump back on when the run is completed by choosing “stop” on the Foxy 200. Choose “run” on the ATS pumps. Adjust the pump speed (Section 9.17.1). Recap the chart recorder pen when not in use.

14.1.14 Remove the diskette from the benchmate when the run is completed (as signified by a “beep” or when the pump and run light are off).

14.1.15 Place the diskette into the computer. Repeat Section 14.1.6. Choose “sample data.” Choose “review output data.” The diskette holds up to two benchmate runs (output1.dat and output2.dat). Choose the correct data output file. Choose “*I*.” Choose “print.” Tape the printout into the run logbook (Attachment 22.7 and 22.14).

NOTE: *The diskettes only hold two output files. They overwrite the previous ones.*

14.1.16 After the GPC run is completed, concentrate on the turbovap.

14.1.16.1 Semi-volatile soil extracts – Transfer the contents with MeCl to 1 mL pre-marked crimp cap vials (Section 9.4.2). Cap. Label. Complete the run logbook. Make a copy of the GPC run logbook page onto salmon paper. Attach it to the copy of the ASE run logbook page.

14.1.16.2 Pesticide/PCB soil extracts – Transfer the contents with hexane to 2 mL pre-marked screw cap vials (Section 9.24.2). Mix. Transfer approximately 0.5 mL to unmarked 2 mL crimp cap vials (Section 9.4). Cap the vials and tubes. Label the vials and tubes. Complete the run logbook. Mark the meniscus on the screw cap tubes. Label. Store the tubes in the refrigerator located in Room 302. Make a copy of the GPC run logbook page onto salmon paper. Attach it to the copy of the ASE run logbook page.

14.1.16.3 PCB (DRO) extracts – Transfer the contents with hexane to 1 mL pre-marked crimp cap vials (Section 9.4), cap, and label. Make a copy of the GPC run logbook page onto salmon paper and attach to copy of the ASE run logbook page.

14.1.16.4 Semi-volatile oil extracts – Transfer the contents to 5 mL pre-marked tubes (section 9.24.2). Mix. Transfer 1.0 mL to 1 mL pre-marked crimp cap vials (section 9.4.2). Cap the vials and tubes. Label. Complete the run logbook. Make a copy of the GPC run logbook page onto salmon paper. Attach it to the copy of the ASE run logbook page.

14.1.16.5 Pesticide/PCB oil extracts – Transfer the contents with Hexane to 2 mL pre-marked screw cap vials (section 9.24.2). Mix. Follow SOP 402 for silica gel cleanup.

14.1.16.6 Place the extracts into corresponding refrigerators or freezers. Place the paperwork in the appropriate bins.

14.2 OI analytical GPC

NOTE: *The OI GPC is used exclusively for cleanup of water extracts for pesticide/PCB analysis.*

14.2.1 Follow Section 13.4.1 and Section 13.4.2.

14.2.2 Turn the detector on (Section 9.14.2). The green “ready” light indicates when the detector is warmed up. Make sure the pen is inserted in the recorder and that the cap is off. Check to make sure that the sensitivity is set at 0.05. Check to make sure that the chart speed is set at 3 cm/hr.

14.2.3 Follow Section 13.4.4 through Section 13.4.6.

14.2.4 Sequence creation

14.2.4.1 Open the sequence folder on the left side of the “WinSEP” screen.

14.2.4.2 Double click on a recent batch.

14.2.4.3 Save this sequence using the new batch number.

14.2.4.4 Change the sample IDs. (See Attachment 22.17) Verify that all other information is correct. Check to make sure that the correct method is listed. (See Attachment 22.13)

14.2.4.5 Choose “save.”

14.2.5 Open the “status” window. Choose the batch from the “sequence name” pull down menu.

14.2.6 Choose “equilibrate” in the “status” window. Equilibration takes approximately 30 minutes. The pressure should be approximately 250 psig and can be checked in the “status” window.

14.2.7 Sample preparation

14.2.7.1 Water samples are extracted according to internal procedure SOP 405 and are brought to 10 mL in methylene chloride.

14.2.7.2 Attach an acrodisc filter (Section 9.20) to a syringe (Section 9.21).

14.2.7.3 Pour the sample extract into the syringe. Filter into a culture tube (Section 9.1).

14.2.7.4 Transfer the label to the new tube.

14.2.7.5 Filter all extracts in the batch including the MPB and the LCS.

14.2.7.6 Cover the tubes with aluminum foil. Secure with tape.

14.2.7.7 Mark the meniscus on each tube with a sharpie.

14.2.7.8 Place the filtered extracts in sample trays according to the sequence file (Section 14.2.4). Tape the tubes together in the trays to prevent them from moving.

14.2.8 Rinse 50 mL turbovap tubes with methylene chloride. Label them according to the sequence file (Section 14.2.4). Place them in the collection tray.

14.2.9 Choose “start” in the “status” window when equilibration has finished and when the baseline looks free of interferences.

14.2.10 When the run is completed, the pump will turn off automatically. Recap the chart recorder pen when not in use.

14.2.11 After the GPC run is complete, (See Attachment 22.15) concentrate on the turbovap to <1.0 mL. Complete the run logbook. (See Attachment 22.7) Make a copy of the GPC run logbook page onto salmon paper. Attach it to the copy of the water extraction logbook page.

14.2.11.1 Rinse the walls of the turbovap down with 10 mL of hexane. Return to the turbovap. Concentrate to <1.0 mL. Perform the florisil procedure (see internal procedure SOP 403). Complete the florisil run logbook. Make a copy of the florisil run logbook page onto yellow paper. Attach it to the other two logbook copies.

14.2.11.2 Place the extracts into corresponding refrigerators or freezers. Place the paperwork in the appropriate bins.

14.3 J2 Scientific GPC

14.3.1 Follow Section 13.5.1 through 13.5.7.

14.3.2 Sequence creation

14.3.2.1 Open the sequence window (See Attachment 22.20) by clicking the sequence button.

14.3.2.2 In “Designation (ID)” type in the sample number for each sample, beginning with “Rinse#1,” followed by the sample numbers, and ending the sequence with “Rinse #2.” If two batches are run, place “Rinse #2” between the last sample of the first batch, and the first sample of the second batch, and run “Rinse #3” at the end.

14.3.2.3 Change “Sample Type” to method selected for cleanup of samples.

14.3.2.4 Verify that all other information is correct. Check to make sure that the correct method is listed. (See Attachment 22.19).

14.3.2.5 Choose "save." Save as sample batch number.

14.3.3 Sample preparation

14.3.3.1 Water samples are extracted according to internal procedure SOP 405 and are brought to 10 mL in methylene chloride. Soil samples are extracted according to internal procedure SOP 401 and are brought up to 10 mL in methylene chloride.

14.3.3.2 Attach an acrodisc filter (Section 9.20) to a syringe (Section 9.21).

14.3.3.3 Pour the sample extract into the syringe. Filter into a culture tube (Section 9.1).

14.3.3.4 Transfer the label to the new tube.

14.3.3.5 Filter all extracts in the batch including the MPB and the LCS/D.

14.3.3.6 Cover the tubes with aluminum foil. Secure with tape.

14.3.3.7 Mark the meniscus on each tube with a sharpie.

14.3.3.8 Place the filtered extracts in sample trays according to the sequence file (Section 14.3.2). Tape the tubes together in the trays to prevent them from moving.

14.3.4 For pesticide/PCB samples – rinse 200 or 50 mL turbovap tubes with methylene chloride. Label them according to the sequence file (Section 14.3.2). Place them in the collection tray.

14.3.5 For semi-volatile samples – rinse 200 mL turbovap tubes with methylene chloride. Label them according to the sequence file (Section 14.3.2). Place them in the collection tray.

14.3.6 Click the Run Sequence button.

14.3.7 When the run is completed, the pump will turn off automatically.

14.3.8 Create run log printout.

14.3.8.1 After sequence has run, Click File -> Export Sequence report to File. Save sequence report in the appropriate report section (i.e. C:/Program Files/J2 Scientific/J2#63/Reports).

14.3.8.2 Go to desktop and click reports folder for appropriate unit, #63 or #64.

14.3.8.3 Find saved report, open. When prompted for file conversion, click yes to open report. (See Attachment 22.21)

14.3.8.4 Highlight and delete extraneous information at top of report. Highlight run log and change font to size 8. Go to File ->Page Layout and click to change to Landscape layout.

14.3.8.5 Click print.

14.3.8.6 On top of printout, write which J2 unit was used, date, and initial. At bottom of printout, write "Dump," "Collect" and "Rinse." Write the appropriate time in minutes for each function. Record final volume.

14.3.9 For water pesticide/PCB samples – after the GPC run is complete, place foil over each turbovap tube. Evaporate on the turbovap to <1 mL. Make a copy of the GPC run logbook (See Attachment 22.7) page onto salmon paper. Attach it to the copy of the water extraction logbook.

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14.3.9.1 Rinse the walls of the turbovap down with 10 mL of hexane. Return to the turbovap. Concentrate to <1.0 mL. Perform the florisil procedure (see internal procedure SOP 403). Complete the florisil run logbook. Make a copy of the florisil run logbook page onto yellow paper. Attach it to the other two logbook copies.

14.3.9.2 Place the extracts into corresponding refrigerators or freezers. Place the paperwork in the appropriate bins.

14.3.10 For soil samples – After the GPC run is completed add 30 mL hexane to the Turbovap tube. Place foil over each turbovap tube. Concentrate on the turbovap.

14.3.10.1 Semi-volatile extracts – Transfer the contents with MeCl to 1 mL pre-marked crimp cap vials (Section 9.4). Cap. Label. Make a copy of the GPC run logbook page onto salmon paper. Attach it to the copy of the ASE run logbook page.

14.3.10.2 Pesticide/PCB extracts – Transfer the contents with hexane to 2 mL pre-marked screw cap vials (Section 9.24). Mix. Transfer approximately 0.5 mL to unmarked 2 mL crimp cap vials (Section 9.4). Cap the vials and tubes. Label the vials and tubes. Mark the meniscus on the screw cap tubes and vials. Label. Store the tubes in the refrigerator located in Room 302. Make a copy of the GPC run logbook page onto salmon paper. Attach it to the copy of the ASE run logbook page.

14.3.10.3 PCB (DRO) extracts – Transfer the contents with hexane to 1 mL pre-marked crimp cap vials (Section 9.4), cap, and label. Make a copy of the GPC run logbook page onto salmon paper and attach to copy of the ASE run logbook page.

14.3.10.4 Place the extracts into corresponding refrigerators or freezers. Place the paperwork in the appropriate bins.

14.4 Glassware cleaning.

14.4.1 Glassware is rinsed with MeCl. It is then placed in the dishwasher. Glassware is also rinsed with MeCl prior to use. Run dishwasher as in attachment 22.23.

15.0 Calculations

15.1 Resolution – See Attachment 22.12 for peak identification of calibration solution compounds. Resolution must be calculated between:

Corn oil and bis (2-ethylhexyl) phthalate
Bis (2-ethylhexyl) phthalate and methoxychlor
Methoxychlor and perylene
Perylene and sulfur

% resolution = (A/B) x 100

A = height of the valley between the peaks.
B = height of the smaller peak.

15.1.1 Measurements for A and B can be found in Attachment 22.8.

15.2 Windows – Windows are determined from the calibration solution analysis in Section 13.

15.2.1 Semi-volatile Windows – Extract is collected just prior to bis (2-ethylhexyl) phthalate and ends just prior to the start of the sulfur peak.

15.2.2 Pesticide/PCB windows – Extract is collected just prior to methoxychlor and ends just prior to the start of the sulfur peak.

15.3 Retention time shift (RTS) – RTS is determined between the newest calibration and the previous calibration.

RTS = ((new RT – previous RT)/previous RT) x 100

RT = retention time.

16.0 Method Performance

16.1 Each MDL study must be labeled with the analyst's name and the date.

16.2 Each IDOA study must be labeled with the analyst's name and the date. A copy must be placed into the analyst's training file.

17.0 Pollution Prevention

17.1 This method uses large quantities of methylene chloride. The waste that is produced is collected for disposal and labeled as chlorinated waste. See SOP 111.

17.2 For information concerning pollution prevention which may be applicable to laboratory operations, consult Less is Better: Laboratory Chemical Management for Waste Reduction available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, DC 20036.

18.0 Data Assessment

18.1 Calibration solution

18.1.1 Check to make sure that all five compounds are present.

18.1.2 Check to make sure that the peaks are symmetrical.

18.1.3 RTS

18.1.3.1 The retention times for bis (2-ethylhexyl) phthalate and perylene must not shift more than 5% from the previous calibration.

18.1.4 Resolution

18.1.4.1 The following resolution must be attained with the calibration solution, or corrective action must be taken:

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Corn oil and bis (2-ethylhexyl) phthalate	>85%
Bis (2-ethylhexyl) phthalate and methoxychlor	>85%
Methoxychlor and perylene	>85%
Perylene and sulfur	>90%

19.0 Corrective Actions

19.1 Quality assurance/quality control (QA/QC) – If the QA/QC is not met and if it is determined to be attributed to the extraction or cleanup, samples may be repeated through this cleanup process.

19.2 Procedure if resolution criteria is not met.

19.2.1 Disconnect the guard column. Replace it as necessary.

19.2.2 Reverse the flow through the analytical column dumping it directly into a waste container.

19.2.3 Set the pump flow at 2.5 mL/min.

19.2.4 Place 6 culture tubes containing 1-chlorobutane (Section 10.9) onto the GPC. Run as samples.

19.2.5 Reconnect the guard column to the analytical column. Restore the proper flow direction.

19.2.6 Set the pump flow at 5.0 mL/min.

19.2.7 Equilibrate for at least one hour.

19.2.8 Recalibrate as in Section 13.

19.2.9 If resolution fails again, repeat section 19.2.1 thru 19.2.8.

19.2.10 The column(s) may need to be replaced if resolution continues to fail.

NOTE: *Retain all used columns for possible repacking.*

19.3 RTs

19.3.1 If RTs shift more than 5% and no obvious reason is apparent (i.e.: new column or new lines on the GPC), check to be sure flow is between 4.5 and 5.5 mL/minute.

19.3.2 If flow is not correct, check for leaks or plugs.

19.3.3 If flow is correct, run another calibration. If it agrees with the new RTs, change windows and run samples.

19.4 Flow must always be measured when the columns are changed or any other major instrument change.

20.0 Waste Management

20.1 The Laboratory Safety Officer arranges for proper waste disposal. See SOP 111.

21.0 References

21.1 United States Code of Federal Regulations, Title 40, U.S. Government Printing Office, Washington, D.C., Appendix A and B.

21.2 Test Methods for Evaluating Solid Waste, U.S. Environmental Protection Agency, Document SW-846, Third Edition, 1986, Final Update III, December 1996.

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22.0 Attachments

22.1 Stock standard logbook page.

22.2 Semi-volatile standard logbook page.

22.3 Benchmate calibration program.

22.4 Benchmate clean up program 3.10
22.4.1 Benchmate clean up program 2.5.

22.5 Benchmate sample ID example.

22.6 Foxy program.

22.7 GPC run logbook page.

22.8 GPC calibration logbook page.

22.9 Hexane MSDS.

22.10 Methylene chloride MSDS.

22.11 1-chlorobutane MSDS.

22.12 Calibration solution chromatogram.

22.13 OI program.

22.14 Benchmate example of output data.

22.15 OI example of output data.

22.16 Benchmate 10 mL start procedures.

22.17 OI sample ID example.

22.18 OI calibration sequence method.

22.19 J2 Method Data page example

22.20 J2 Sequence Setup Example

22.21 J2 Example of Output data

22.22 J2 Example of Calibration Data

22.23 Lancer Dishwasher Procedure

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22.24 Acetone MSDS

22.25 Methyl Alcohol MSDS

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Signature Page

APPROVED BY _____ DATE 2/26/07
CHIEF, LABORATORY SERVICES SECTION

APPROVED BY _____ DATE _____
COMPLIANCE OFFICER

APPROVED BY _____ DATE 2/22/07
UNIT MANAGER

APPROVED BY _____ DATE 2/21/07
SECOND SHIFT MANAGER

APPROVED BY _____ DATE 2/21/07
ANALYST

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MDEQ-ENVIRONMENTAL LAB
STOCK TRACKING LOG

Environmental Lab Tracking Number	QTY (each)	Filley Tracking Number	Date Received (at lab)	Receiver Initials	Chemical,Analyte, Reagent or Solvent	Vendor and Catalog #	PO #	Manufacturer and Lot Number	Expiration Date
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
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EL-									
EL-									

ATTACHMENT 22.1

FILE:ST-STD
12/30/98

MDEQ-ENVIRONMENTAL LAB
SEMI-VOLATILE ORGANIC STANDARDS

SVOC Tracking Number	Date Prepared	EL or SVOC Tracking Number	Analyte or Mix	Preparers Initials	Solvent	Solvent Tracking Number	Initial Concentration*	Initial Volume/Wt Standard*	Final Volume*	Final Concentration*	Expiration Date
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											

ATTACHMENT 22.2

FILE:SV-STD
12/30/98

Zymark BenchMate 2.5

Zymark BenchMate Procedure : DUAL INJECTOR CALIBARTION PROGRAM

Step 1 : Add 9 ml of MeCl2 1
Step 2 : Vortex for 5 seconds at speed 2
Step 3 : Wash syringe with 10 ml of MeCl2 1
Step 4 : Wash LC injector with 10 ml of MeCl2 1
Step 5 : Inject sample on Alternate LC with run time of 5 minutes
Step 6 : Wash syringe with 10 ml of MeCl2 1
Step 7 : Wash LC injector with 10 ml of MeCl2 1
Step 8 : END

SETUP PARAMETERS - 1

SAMPLE TUBE
Initial Volume: 1.00 ml

GRAVIMETRIC PARAMETERS
Gravimetric on: Y Y=YES N=NO
Tolerance: 20%
Tare Sample Tubes: N Y=YES N=NO

FLOW RATES
Aspirate: 0.50 ml/sec
Dispense: 1.00 ml/sec
Internal Std: 0.12 ml/sec
Mix: 1.50 ml/sec
Filter: .210 ml/sec
Air Push: 0.15 ml/sec

RACK PARAMETERS
LV Tube Numbering: Y Y=YES N=NO
Reset to Sample 1: N Y=YES N=NO

DISPENSING
Liquid driven: Y Y=YES N=NO

AUTOWASH PARAMETERS
Int Vol: 0.00 ml
Sample Vol: 0.00 ml

SETUP PARAMETERS - 2

SPE PARAMETERS
Cond Flow: 0.25 ml/sec
Load Flow: 0.15 ml/sec
Rinse Flow: 0.25 ml/sec
Elute Flow: 0.15 ml/sec
Push Delay: 5 sec
Air Factor: 1.0
Save Columns: N Y=YES N=NO

LC PARAMETERS
Inject Load Vol: 10.0 ml
Calibrate every: 0 samples
Calib Replicates: 1
Calibration Reagent: 1
Calib Wash Vol: 0.00 ml

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Zymark BenchMate Procedure : DUAL INJECTOR CALIBARTION PROGRAM

NAME REAGENTS

Reagent 1 : MeCl2 1 ; Density : 1.3170
Reagent 2 : IPA 2 ; Density : 0.0037
Reagent 3 : Hexane 3 ; Density : 0.6600
Reagent 4 : REAGENT 4 ; Density : 1.0000
Reagent 5 : REAGENT 5 ; Density : 1.0000
Reagent 6 : REAGENT 6 ; Density : 1.0000

NAME INTERNAL STANDARD

Standard : INTERNAL STD ; Density : 1.0000

BenchMate Table Setup

THE ESTIMATED TIME FOR ONE SAMPLE IS: 14.4 MINUTES.

NO FILTERS ARE USED.
NO SPE COLUMNS ARE USED.

Tubes will be processed starting at Rack 1 position 1,
through all Racks until an empty position is encountered.

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Zymark BenchMate 3.10

Zymark BenchMate Procedure : CALIB #16 WITH PAUSE

Step 1 : Add 9 ml of MeCl2
Step 2 : Vortex for 3 seconds at speed 2
Step 3 : Wash syringe with 10 ml of MeCl2
Step 4 : Wash LC injector with 10 ml of MeCl2
Step 5 : Inject sample on LC with run time of 5 minutes
Step 6 : Wash LC injector with 10 ml of MeCl2
Step 7 : Wash syringe with 10 ml of MeCl2
Step 8 : Pause for 22 minutes
Step 9 : END

SETUP PARAMETERS

SAMPLE TUBE
Initial Volume: 1.00 ml

GRAVIMETRIC PARAMETERS
Gravimetric on: Y Y=YES N=NO
Tare Sample Tubes: N Y=YES N=NO

FLOW RATES
Aspirate: 0.50 ml/sec
Dispense: 0.25 ml/sec
Internal Std: 0.12 ml/sec
Mix: 1.50 ml/sec
Air Push: 0.15 ml/sec

RACK PARAMETERS
LV Tube Numbering: Y Y=YES N=NO
Reset to Sample 1: N Y=YES N=NO

FILTER PARAMETERS
Filter Flow Rate: 0.21 ml/sec
Filtrate Confirm: N Y=YES N=NO
Filtrate Density: 1.00 g/ml
Minimum Amount: 0%

DISPENSING
Liquid driven: Y Y=YES N=NO

AUTOWASH PARAMETERS
Rinse Reagent: 1
Rinse Vol: 0.00 ml
Purge Vol: 0.00 ml

SETUP SPE PARAMETERS

SPE PARAMETERS
Cond Flow: 0.25 ml/sec
Aspirate Flow: 0.50 ml/sec
Load Flow: 0.15 ml/sec
Rinse Flow: 0.25 ml/sec
Elute Flow: 0.15 ml/sec
Push Delay: 5 sec
Air Factor: 1.0
Save Columns: N Y=YES N=NO
Save Samples: N Y=YES N=NO

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SETUP LC PARAMETERS

LC PARAMETERS
Inject Std Vol: 10.0 ml
Inject Delay Time: 99 sec
Loop in Load Pos: Y Y = YES N = NO
Inject Loop Time: 99 sec

ENCORE PARAMETERS
Load disk methods: Y Y = YES N = NO
Lock pump keypad: Y Y = YES N = NO
Initialization Time: 0 min
Equilibration Time: 0 min
Shutdown method: 0

LC CALIBRATION STANDARDS
Calibrate every: 0 samples
INJECT START DURING END
LC Std 1: 0 0 0
LC Std 2: 0 0 0
LC Std 3: 0 0 0
LC Std 4: 0 0 0
LC Std 5: 0 0 0
LC Std 6: 0 0 0
Calib Wash Vol: 0.00 ml
Calib Wash Reagent: 1

SETUP EasyFill PARAMETERS

EasyFill PARAMETERS
EasyFill vial type: 2
Sense vials: Y Y = YES N = NO
External needle wash: Y Y = YES N = NO

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Current BenchMate Procedure : CALIB #16 WITH PAUSE

NAME REAGENTS

Agent 1 : Acetone ; Density : 1.3170
Reagent 2 : IPA ; Density : 0.8037
Reagent 3 : Hexane ; Density : 0.6600
Reagent 4 : REAGENT 4 ; Density : 1.0000
Reagent 5 : REAGENT 5 ; Density : 1.0000
Reagent 6 : REAGENT 6 ; Density : 1.0000

NAME INTERNAL STANDARD

Standard : INTERNAL STD ; Density : 1.0000

BenchMate Table Setup

THE ESTIMATED TIME FOR ONE SAMPLE IS: 38.7 MINUTES.

NO FILTERS ARE USED.
NO SPE COLUMNS ARE USED.

Tubes will be processed starting at: Rack 1 position 1.
through all Racks until an empty position is encountered.

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Zymark BenchMate 3.10

Zymark BenchMate Procedure : 10ML CLEANUP WITH FILTER

Step 1 : Wash syringe with 10 ml of MECL2
Step 2 : Wash LC injector with 10 ml of MECL2
Step 3 : Prewet filter with 10 ml of ACETONE
Step 4 : Prewet filter with 10 ml of MEOH
Step 5 : Prewet filter with 10 ml of MECL2
Step 6 : Prewet filter with 10 ml of MECL2
Step 7 : Vortex for 10 seconds at speed 2
Step 8 : Filter 9.8 ml of sample into next tube
Step 9 : Rinse filter holder with 10 ml of ACETONE
Step 10 : Rinse filter holder with 10 ml of MEOH
Step 11 : Rinse filter holder with 10 ml of MECL2
Step 12 : Rinse filter holder with 10 ml of MECL2
Step 13 : Wash syringe with 10 ml of MECL2
Step 14 : Inject sample on LC with run time of 9 minutes
Step 15 : Pause for 3 minutes
Step 16 : Wash syringe with 10 ml of ACETONE
Step 17 : Wash syringe with 10 ml of MEOH
Step 18 : Wash syringe with 10 ml of MECL2
Step 19 : Wash LC injector with 10 ml of ACETONE
Step 20 : Wash LC injector with 10 ml of MEOH
Step 21 : Wash LC injector with 10 ml of MECL2
Step 22 : Wash LC injector with 10 ml of MECL2
Step 23 : END

SETUP PARAMETERS

SAMPLE TUBE
Initial Volume: 10.0 ml

GRAVIMETRIC PARAMETERS
Gravimetric on: Y Y=YES N=NO
Tare Sample Tubes: N Y=YES N=NO

FLOW RATES
Aspirate: 0.50 ml/sec
Dispense: 1.00 ml/sec
Internal Std: 0.12 ml/sec
Mix: 1.50 ml/sec
Air Push: 0.15 ml/sec

RACK PARAMETERS
LV Tube Numbering: Y Y=YES N=NO
Reset to Sample 1: N Y=YES N=NO

FILTER PARAMETERS
Filter Flow Rate: 0.10 ml/sec
Filtrate Confirm: N Y=YES N=NO
Filtrate Density: 1.00 g/ml
Minimum Amount: 0%

DISPENSING
Liquid driven: Y Y=YES N=NO

AUTOWASH PARAMETERS
Rinse Reagent: 1
Reagent Vol: 1.00 ml
Sample Vol: 0.20 ml

SETUP SPE PARAMETERS

SPE PARAMETERS
Cond Flow: 0.25 ml/sec
Aspirate Flow: 0.25 ml/sec
Load Flow: 0.15 ml/sec
Rinse Flow: 0.25 ml/sec
Elute Flow: 0.15 ml/sec
Push Delay: 5 sec

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Air Factor: 1.0
Save Columns: N Y-YES N-NO
Save Samples: N Y-YES N-NO

SETUP LC PARAMETERS

LC PARAMETERS
Inject Sip Vol: 8.50 ml
Inject Delay Time: 99 sec
Loop in Load Pos: N Y-YES N-NO
Inject Loop Time: 99 sec

LC CALIBRATION STANDARDS
Calibrate every: 0 samples
INJECT START DURING END
LC Std 1: 0 0 0
LC Std 2: 0 0 0
LC Std 3: 0 0 0

ENCORE PARAMETERS
Load disk methods: Y Y-YES N-NO
Lock pump keypad: Y Y-YES N-NO
Initialization Time: 0 min
Equilibration Time: 0 min
Shutdown method: 0

LC Std 4: 0 0 0
LC Std 5: 0 0 0
LC Std 6: 0 0 0
Calib Wash Vol: 0.00 ml
Calib Wash Reagent: 1

SETUP EasyFill PARAMETERS

EasyFill PARAMETERS
EasyFill vial type: 2
Sense vials: Y Y-YES N-NO
External needle wash: Y Y-YES N-NO

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Zymark BenchMate Procedure : 10ML CLEANUP WITH FILTER

NAME REAGENTS

Reagent 1 : MECL2 ; Density : 1.3255
Reagent 2 : ACETONE ; Density : .78800
Reagent 3 : MEOH ; Density : 0.7866
Reagent 4 : REAGENT 4 ; Density : 1.0000
Reagent 5 : REAGENT 5 ; Density : 1.0000
Reagent 6 : REAGENT 6 ; Density : 1.0000

NAME INTERNAL STANDARD

Standard : INTERNAL STD ; Density : 1.0000

BenchMate Table Setup

THE ESTIMATED TIME FOR ONE SAMPLE IS: 41.9 MINUTES.

PLACE FILTERS IN FILTER DISPENSER.
NO SPE COLUMNS ARE USED.

Samples will be processed starting at Rack 1 position 1,
through position 50, then Rack 2 positions 1 through 50
or until an empty position is encountered.

Final tubes are in Racks 3 and 4.

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Zymark BenchMate 2.5

Zymark BenchMate Procedure : INJECTOR #14 GPC CLEAN-UP(8 MIN PAUSE)

Step 1 : Add 8 ml of MeCl2
Step 2 : Wash syringe with 10 ml of MeCl2
Step 3 : Wash LC injector with 10 ml of MeCl2
Step 4 : Prewet filter with 10 ml of MeCl2
Step 5 : Prewet filter with 10 ml of MeCl2
Step 6 : Prewet filter with 10 ml of MeCl2
Step 7 : Vortex for 10 seconds at speed 2
Step 8 : Filter 9.8 ml of sample into next tube
Step 9 : Rinse filter holder with 10 ml of ACETONE
Step 10 : Wash syringe with 10 ml of MEOH
Step 11 : Wash syringe with 10 ml of MeCl2
Step 12 : Wash syringe with 10 ml of MeCl2
Step 13 : Inject sample on LC 1 with run time of 9 minutes
Step 14 : Wash syringe with 10 ml of ACETONE
Step 15 : Wash syringe with 10 ml of MEOH
Step 16 : Wash syringe with 10 ml of MeCl2
Step 17 : Wash syringe with 10 ml of MeCl2
Step 18 : Wash LC injector with 10 ml of ACETONE
Step 19 : Wash LC injector with 10 ml of MEOH
Step 20 : Wash LC injector with 10 ml of MeCl2
Step 21 : Wash LC injector with 10 ml of MeCl2
Step 22 : END

SETUP PARAMETERS - 1

SAMPLE TUBE
Initial Volume: 2.00 ml

FLOW RATES
Aspirate: 0.50 ml/sec
Dispense: 1.00 ml/sec
Internal Std: 0.12 ml/sec
Mix: 1.50 ml/sec
Filter: 0.21 ml/sec
Air Push: 0.15 ml/sec

GRAVIMETRIC PARAMETERS
Gravimetric on: Y Y=YES N=NO
Tolerance: 20%
Tare Sample Tubes: N Y=YES N=NO

RACK PARAMETERS
LV Tube Numbering: Y Y=YES N=NO
Reset to Sample 1: N Y=YES N=NO

DISPENSING
Liquid driven: Y Y=YES N=NO

AUTOWASH PARAMETERS
Reagent Vol: 0.00 ml
Sample Vol: 0.00 ml

SETUP PARAMETERS - 2

SPE PARAMETERS
Cond Flow: 0.25 ml/sec
Load Flow: 0.15 ml/sec
Rinse Flow: 0.25 ml/sec
Elute Flow: 0.15 ml/sec
Push Delay: 5 sec
Air Factor: 1.0
Save Columns: N Y=YES N=NO

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LC PARAMETERS
Inject Load Vol: 8.00 ml
Calibrate every: 0 samples
Calib Replicates: 1
Calibration Reagent: 1
Calib Wash Vol: 0.00 ml

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Zymark BenchMate Procedure : INJECTOR #14 GPC CLEAN-UP(8 MIN PAUSE)

NAME REAGENTS

Reagent 1 : MeCl2 ; Density : 1.3255
Reagent 2 : ACETONE ; Density : 0.7880
Reagent 3 : IPA ; Density : 0.7850
Reagent 4 : MEOH ; Density : 0.7915
Reagent 5 : HEXANE ; Density : 0.6600
Reagent 6 : DI H2O ; Density : 1.0000

NAME INTERNAL STANDARD

Standard : INTERNAL STD ; Density : 1.0000

BenchMate Table Setup

THE ESTIMATED TIME FOR ONE SAMPLE IS: 40.8 MINUTES.

PLACE FILTERS IN FILTER DISPENSER.
NO SPE COLUMNS ARE USED.

Samples will be processed starting at Rack 1 position 1, through position 50, then Rack 2 positions 1 through 50 or until an empty position is encountered.

Final tubes are in Racks 3 and 4.

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Heading 1: GPC CLEAN-UP #14
Heading 2: BATCH 1532 PCB PPS
Heading 3: TM
'al Vol: 2
Entered: Wed Jun 26 11:45:00 2002

Position	Sample ID	Initial Wt
1	MPB 1532	0.0000
2	LCS 1532	0.0000
3	MS 1532	0.0000
4	MSD 1532	0.0000
5	06 194 01	0.0000
6	06 218 21	0.0000
7	06 218 22	0.0000
8	06 218 24	0.0000
9	06 218 26	0.0000
10	06 218 27	0.0000
11	06 218 28	0.0000
12	06 218 29	0.0000
13	06 218 32	0.0000
14	06 218 33	0.0000
15	06 207 01	0.0000
16	06 207 02	0.0000
17	06 207 03	0.0000
18	RINSE	0.0000

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Attachment 22.6

FOXY COLLECTION PARAMETERS

Settings are the same for foxy units on all Benchmates unless noted. Press button under bold choice or hit enter.

Edit A (B for #14)
TIME/DROP/VOLTJME
TIME
Time windows Yes

Programming for **RACK 1** (front)
Tube size: custom A **ENTER** Tubes/rack=9 **ENTER**
Custom rack: Max # tubes/row=3 **ENTER**
Drop former will move to position of tubes #1 and #9. Check to see positioning is correct.
Example coordinates are approximate.
1 F to B=1421 L to R=0309 **ENTER**
9 F to B=0924 L to R=1010 **ENTER**
Peak = Rack 1&2 Nonpeak=Drain **ENTER**
Peak Fraction size=30.00 **ENTER**
Rack 2 **Different** .
Tube size: custom B **ENTER** Tubes/rack=9 **ENTER**
Custom rack: # tubes per row=3 **ENTER**
Drop former will move to position of tubes #10 and #18. Check to see positioning is correct.
Example coordinates are approximate.
1 F to B=0600 L to R=03l3 **ENTER**
9 F to B=0090 L to R=1000 **ENTER**
Peak Fraction size=30.00 **ENTER**
PEAK
Time Windows Only **ENTER**
Times are approximate — see chart in front of FOXY 200
BNA— 12:00—23:00
Pest/PCB— 13:30—22:50
Exit

Page 2
RESTART
MOMENTARY
# Of collections before advancing=1 **ENTER**
Restart=**NEXT TUBE**
Select program for restart A (B for #14)

Page 3
Technical
Pattern = **ALWAYS LEFT TO RIGHT** or
= ONE DIRECTION DEPENDING ABOUT FOXY USED
Drop Wait=
Should drop former wait? YES

STOP (Red key)
If changes were made, press Save As A full (B for #14)
If no changes were made, press Ready A (B for #14)
If you want to check program, press Quick Review
More changes can be made in Edit A (B for #14)
RUN A (or B) to run.

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MDEQ-Environmental Lab
GPC Cleanup Logbook- Organics

Method: 3640
Date:_____
Analyst:_____

MeCl #

Hexane #

Benchmate #	Window (minutes)	Batch #	Fraction Size (minutes)
14*			
15*			
16			

Sample ID		Final Volume (mL)	Comments

COMMENTS

NOTE: 2mL of sample extract is diluted to 10mL and then 5mL is injected onto the GPC Column.

Note: The final solvent for Pesticide and PCB analysis is hexane.

*Dual injection occurs when #14 & #15 are both circled.(Then odd #'s = #14; even #'s = #15)

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MDEQ-Environmental Lab
GPC Cleanup Logbook- Organics
Instrument # 41

Analyst________

Method 3640

Minutes	
Dump Time:	
Collect Time:	
Wash Time:	

MeCl #

Hexane #

Date	Start Time	Injection time	End Time	Results	Method	Sample Vial	Sample ID	Final Volume (mL)	Comments

NOTE: 10mL of filtered sample extract is placed in the GPC tube. 5mL is injected onto the GPC Column.

Comments:

NOTE: The final solvent for Pesticide & PCB analysis is Hexane.

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Method: 3640
Date:_____
Analyst:_____

MDEQ-Environmental Lab
GPC Calibration Logbook
Organics
Instrument # 16

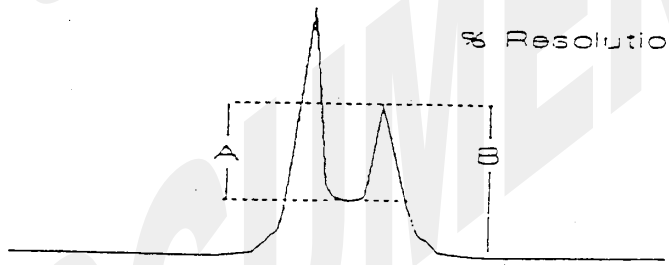
Noise Filter: _____
Peak Separation _____
Sensitivity _____
Chart Speed _____

Flow Rate mL/min _____
GPC Calibration Solution # _____
Volume Injected (mL) _____
Room Temperature _____

Peak #	Compound	Peak Start Time (minutes)	Peak Time (minutes)	Valley After Peak (minutes)	
1	Corn Oil				
2	Bis(2-ethyihexyl) phthalate				
3	Methoxychlor				
4	Perylene				
5	Sulfur				

	Resolution Criteria	A = depth of valley (mm)	B = height of shorter peak (mm)	Calculated Resolution (%)	Acceptable (yes/no initials)
	Corn Oil & Phthalate (>85%)				
	Phthalate & Methoxychlor (>85%)				
	Methoxychlor & Perylene (>85%)				
	Perylene & Sulfur (>90%)				

$$\% \text{ Resolution} = \frac{A}{B} \times 100$$



	BNA Collect Start Time (min)	BNA Collect End Time (min)	Pesticide/PCB Collect Start Time (min)	Pesticide/PCB Collect End Time (min)	Acceptable (yes/no & initials)
		Retention time shift of phthalate from last calibration (not more than +/- 5%)	Retention time shift of perylene from last calibration (not more than +/- 5%)		

$$\text{Retention Time Shift} = \frac{(\text{New RT} - \text{Last RT}) \times 100}{\text{Last RT}}$$

COMMENTS

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MSDS Number: **H2381** * * * * * *Effective Date:* ***08/10/04*** * * * * * *Supercedes:* ***11/02/01***

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

J.T.Baker®

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. And Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

HEXANE

1. Product Identification

Synonyms: Hexanes,Normal Hexane; Hexyl Hydride; Hexane 95%
CAS No.: 110-54-3 (n-hexane)
Molecular Weight: 86.18
Chemical Formula: CH3(CH2)4CH3 n-hexane
Product Codes: 9262, 9304, 9308, N168

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Hexane	110-54-3	85 - 100%	Yes
Methylcyclopentane	96-37-7	1 - 2%	Yes
Trace amount of Benzene (10 ppm)	071-43-2	*	No

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3. Hazards Identification

Emergency Overview

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 2 - Moderate
Flammability Rating: 3 - Severe (Flammable)
Reactivity Rating: 0 - None
Contact Rating: 2 - Moderate
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

The health hazards addressed are for the major component: n-hexane.

Inhalation:
Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Greater exposure may cause muscle weakness, numbness of the extremities, unconsciousness and death.

Ingestion:
May produce abdominal pain, nausea. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms expected to parallel inhalation.

Skin Contact:
May cause redness, irritation, with dryness, cracking.

Eye Contact:
Vapors may cause irritation. Splashes may cause redness and pain.

Chronic Exposure:
Repeated or prolonged skin contact may defat the skin and produce irritation and dermatitis. Chronic inhalation may cause peripheral nerve disorders and central nervous system effects.

Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance. May affect the developing fetus.

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4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:
Aspiration hazard. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:
Remove any contaminated clothing. Wipe off excess from skin. Wash skin with soap and water for at least 15 minutes. Get medical attention if irritation develops or persists.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

Note to Physician:
BEI=2,5-hexadione in urine, sample at end of shift at workweeks end, 5 mg/g creatine. Also, measure n-hexane in expired air. Analgesics may be necessary for pain management, there is no specific antidote. Monitor arterial blood gases in cases of severe aspiration.

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5. Fire Fighting Measures

Fire:
Flash point: -23C (-9F) CC
Autoignition temperature: 224C (435F)
Flammable limits in air % by volume:
lel: 1.2; uel: 7.7
Extremely Flammable Liquid and Vapor! Vapor may cause flash fire. Dangerous fire hazard when exposed to heat or flame.
Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Contact with oxidizing materials may cause extremely violent combustion.Explodes when mixed @ 28C with dinitrogen tetraoxide. Sensitive to static discharge.
Fire Extinguishing Media:
Dry chemical, foam or carbon dioxide. Water may be ineffective.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Water spray may be used to keep fire exposed containers cool. Vapors can flow along surfaces to distant ignition source and flash back. Vapor explosion hazard exists indoors, outdoors, or in sewers.

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6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB® solvent adsorbent is recommended for spills of this product.

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7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from direct sunlight and any area where the fire hazard may be acute. Store in tightly closed containers (preferably under nitrogen atmosphere). Outside or detached storage is preferred. Inside storage should be in a standard flammable liquids storage room or cabinet. Separate from oxidizing materials. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
N-Hexane [110-54-3]:
-OSHA Permissible Exposure Limit (PEL): 500 ppm (TWA)
-ACGIH Threshold Limit Value (TLV): 50 ppm (TWA), Skin
other isomers of hexane
-ACGIH Threshold Limit Value (TLV): 500 ppm (TWA),1000ppm (STEL)
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for

details.

Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded and engineering controls are not feasible, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. Breathing air quality must meet the requirements of the OSHA respiratory protection standard (29CFR1910.134).

Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

Appearance:
Clear, colorless liquid.

Odor:
Light odor.

Solubility:
Insoluble in water.

Specific Gravity:
0.66

pH:
No information found.

% Volatiles by volume @ 21C (70F):
100

Boiling Point:
ca. 68C (ca. 154F)

Melting Point:
ca. -95C (ca. -139F)

Vapor Density (Air=1):
3.0

Vapor Pressure (mm Hg):
130 @ 20C (68F)

Evaporation Rate (BuAc=1):
9

---

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Heat will contribute to instability.

Hazardous Decomposition Products:

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May produce acrid smoke and irritating fumes when heated to decomposition.

Hazardous Polymerization:

Will not occur.

Incompatibilities:

Strong oxidizers.

Conditions to Avoid:

Heat, flames, ignition sources and incompatibles.

---

11. Toxicological Information

N-Hexane: Oral rat LD50: 28710 mg/kg. Irritation eye rabbit: 10 mg mild. Investigated as a tumorigen, mutagen and reproductive effector.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Hexane (110-54-3)	No	No	None
Methylcyclopentane (96-37-7)	No	No	None
Trace amount of Benzene (10 ppm) (071-43-2)	Yes	No	1

---

12. Ecological Information

Environmental Fate:

When released into the soil, this material may biodegrade to a moderate extent. When released into the soil, this material is not expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material may biodegrade to a moderate extent. When released to water, this material is expected to quickly evaporate. When released into the water, this material is expected to have a half-life between 1 and 10 days. This material has an estimated bioconcentration factor (BCF) of less than 100. This material has a log octanol-water partition coefficient of greater than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material is expected to be readily degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material is expected to have a half-life between 1 and 10 days.

Environmental Toxicity:

No information found.

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13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

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14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: HEXANES
Hazard Class: 3
UN/NA: UN1208
Packing Group: II
Information reported for product/size: 215L

International (Water, I.M.O.)

Proper Shipping Name: HEXANES
Hazard Class: 3
UN/NA: UN1208
Packing Group: II
Information reported for product/size: 215L

---

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Hexane (110-54-3)	Yes	Yes	Yes	Yes
Methylcyclopentane (96-37-7)	Yes	Yes	No	Yes
Trace amount of Benzene (10 ppm) (071-43-2)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

--Canada--

Ingredient	Korea	DSL	NDSL	Phil.
Hexane (110-54-3)	Yes	Yes	No	Yes
Methylcyclopentane (96-37-7)	Yes	Yes	No	Yes
Trace amount of Benzene (10 ppm) (071-43-2)	Yes	Yes	No	Yes

\-------\Federal, State & International Regulations - Part 1\-------

Ingredient	-SARA 302- RQ	TPQ	List	------SARA 313------ Chemical Catg.
Hexane (110-54-3)	No	No	Yes	No
Methylcyclopentane (96-37-7)	No	No	No	No
Trace amount of Benzene (10 ppm) (071-43-2)	No	No	Yes	No

\-------\Federal, State & International Regulations - Part 2\-------

Ingredient	CERCLA	-RCRA- 261.33	8(d)	TSCA-
Hexane (110-54-3)	5000	No	No	
Methylcyclopentane (96-37-7)	No	No	No	
Trace amount of Benzene (10 ppm) (071-43-2)	10	U019	No	

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: Yes Pressure: No
Reactivity: No (Mixture / Liquid)

WARNING:
THIS PRODUCT CONTAINS A CHEMICAL(S) KNOWN TO THE STATE OF CALIFORNIA TO CAUSE CANCER.

Australian Hazchem Code: 3[Y]E
Poison Schedule: None allocated.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

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16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**

Label Hazard Warning:

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.

Label Precautions:

Keep away from heat, sparks and flame.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor or mist.
Avoid contact with eyes, skin and clothing.

Label First Aid:

Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. In all cases call a physician.

Product Use:

Laboratory Reagent.

Revision Information:

No Changes.

Disclaimer:

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Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

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MSDS Number: **M4420** * * * * * *Effective Date:* ***10/06/05*** * * * * *Supercedes:* ***05/14/03***

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. And Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

METHYLENE CHLORIDE

1. Product Identification

Synonyms: MC; Dichloromethane (DCM); Methylene dichloride; Methylene bichloride; Methane dichloride
CAS No.: 75-09-2
Molecular Weight: 84.93
Chemical Formula: CH2Cl2
Product Codes: 9235, 9264, 9266, 9295, 9315, 9324, 9329, 9330, 9348, 9350, 9965, Q480

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Methylene Chloride	75-09-2	> 99%	Yes

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3. Hazards Identification

Emergency Overview

WARNING! HARMFUL IF SWALLOWED, INHALED OR ABSORBED THROUGH SKIN. AFFECTS CENTRAL NERVOUS SYSTEM, LIVER, CARDIOVASCULAR SYSTEM, AND BLOOD. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. SUSPECT CANCER HAZARD. MAY CAUSE CANCER. Risk of cancer depends on level and duration of exposure.

SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Cancer Causing)
Flammability Rating: 1 - Slight
Reactivity Rating: 2 - Moderate
Contact Rating: 3 - Severe
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: Blue (Health)

Potential Health Effects

Inhalation:
Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes formation of carbon monoxide in blood which affects cardiovascular system and central nervous system. Continued exposure may cause increased light-headedness, staggering, unconsciousness, and even death. Exposure may make the symptoms of angina (chest pains) worse.

Ingestion:
May cause irritation of the gastrointestinal tract with vomiting. If vomiting results in aspiration, chemical pneumonia could follow. Absorption through gastrointestinal tract may produce symptoms of central nervous system depression ranging from light headedness to unconsciousness.

Skin Contact:
Causes irritation, redness and pain. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Eye Contact:
Vapors can cause eye irritation. Contact can produce pain, inflammation and temporal eye damage.

Chronic Exposure:
Can cause headache, mental confusion, depression, liver effects, kidney effects,

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bronchitis, loss of appetite, nausea, lack of balance, and visual disturbances. Can cause dermatitis upon prolonged skin contact. Methylene chloride may cause cancer in humans.

Aggravation of Pre-existing Conditions:

Persons with pre-existing skin disorders, eye problems, impaired liver, kidney, respiratory or cardiovascular function may be more susceptible to the effects of this substance.

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4. First Aid Measures

Inhalation:

Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.

Ingestion:

If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Get medical attention immediately.

Skin Contact:

Immediately flush skin with plenty of soap and water for at least 15 minutes while removing contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.

Eye Contact:

Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

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5. Fire Fighting Measures

Fire:

Autoignition temperature: 556C (1033F)
Flammable limits in air % by volume:
lel: 12; uel: 23
Forms flammable vapor-air mixtures above 100C (212F).

Explosion:

Concentrated can be ignited by a high intensity ignition source. Vapor may form flammable mixture in atmosphere that contains a high percentage of oxygen. Sealed containers may rupture when heated.

Fire Extinguishing Media:

Dry chemical, foam or carbon dioxide. Water spray may be used to keep fire exposed containers cool.

Special Information:

In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Combustion by-products include phosgene and hydrogen chloride gases. Structural firefighters' clothing provides only limited protection to the combustion products of this material.

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6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

7. Handling and Storage

Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect against physical damage. Isolate from any source of heat or ignition. Outside or detached storage is recommended. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product. To minimize decomposition, all storage containers should be galvanized or lined with a phenolic coating. This material may corrode plastic and rubber. Wear special protective equipment (Sec. 8) for maintenance break-in or where exposures may exceed established exposure levels. Wash hands, face, forearms and neck when exiting restricted areas. Shower, dispose of outer clothing, change to clean garments at the end of the day. Avoid cross-contamination of street clothes. Wash hands before eating and do not eat, drink, or smoke in workplace. Odor Threshold: 205 - 307 ppm. The odor threshold only serves as a warning of exposure; not smelling it does not mean you are not being exposed.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
Methylene Chloride (Dichloromethane):
- OSHA Permissible Exposure Limit (PEL) -
25 ppm (TWA), 125 ppm (STEL), 12.5 ppm (8-hour TWA - Action Level)
- ACGIH Threshold Limit Value (TLV) -
50 ppm (TWA), A3 - suspected human carcinogen.
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred

because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

Personal Respirators (NIOSH Approved):

If the exposure limit is exceeded, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. The cartridges recommended for this material have a predicted service of less than 30 minutes at concentrations of ten times (10x) the exposure limits. Actual service life will vary considerbly, depending on concentration levels, temperature, humidity, and work rate. This substance has poor warning properties.

Skin Protection:

Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact. Neoprene is a recommended material for personal protective equipment. Natural rubber and polyvinyl chloride ARE NOT recommended materials for personal protective equipment.

Eye Protection:

Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

Other Control Measures:

Do not use closed circuit rebreathing system employing soda lime or other carbon dioxide absorber because of formation of toxic compounds capable of producing cranial nerve paralysis. See OSHA Standard for medical surveillance, record keeping, and reporting requirements for methylene chloride (29 CFR 1910.1052).

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9. Physical and Chemical Properties

Appearance:

Clear, colorless liquid.

Odor:

Chloroform-like odor.

Solubility:

1.32 gm/100 gm water @ 20C.

Specific Gravity:

1.318 @ 25C

pH:

No information found.

% Volatiles by volume @ 21C (70F):

100

Boiling Point:

39.8C (104F)

Melting Point:

-97C (-143F)

Vapor Density (Air=1):

2.9

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Vapor Pressure (mm Hg):
350 @ 20C (68F)
Evaporation Rate (BuAc=1):
27.5

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10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products:
Emits highly toxic fumes of phosgene when heated to decomposition. Decomposes in a flame or hot surface to form toxic gas phosgene and corrosive mists of hydrochloric acid. Carbon dioxide and carbon monoxide may form when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Strong oxidizers, strong caustics, plastics, rubber, nitric acid, water + heat, and chemically active metals, such as aluminum and magnesium powder, sodium, potassium, and lithium. Avoid contact with open flames and electrical arcs. Liquid methylene chloride will attack some forms of plastics, rubber, and coatings.
Conditions to Avoid:
Moisture, heat, flames, ignition sources and incompatibles.

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11. Toxicological Information

Toxicological Data:
Dichloromethane: Oral rat LD50: 1600 mg/kg; inhalation rat LC50: 52 gm/m3; investigated as a tumorigen, mutagen, reproductive effector.
Reproductive Toxicity:
Dichloromethane has been linked to spontaneous abortions in humans.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Methylene Chloride (75-09-2)	No	Yes	2B

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12. Ecological Information

Environmental Fate:
When released into the soil, this material may leach into groundwater. When released

into the soil, this material is expected to quickly evaporate. When released into water, this material may biodegrade to a moderate extent. When released to water, this material is expected to quickly evaporate. This material has a log octanol-water partition coefficient of less than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material may be moderately degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material is expected to have a half-life of greater than 30 days. When released into the air, this material may be removed from the atmosphere to a moderate extent by wet deposition.
Environmental Toxicity:
The LC50/96-hour values for fish are over 100 mg/l. This material is not expected to be toxic to aquatic life.

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13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

---

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: RQ, DICHLOROMETHANE
Hazard Class: 6.1
UN/NA: UN1593
Packing Group: III
Information reported for product/size: 1000L

International (Water, I.M.O.)

Proper Shipping Name: RQ, DICHLOROMETHANE
Hazard Class: 6.1
UN/NA: UN1593
Packing Group: III
Information reported for product/size: 1000L

International (Air, I.C.A.O.)

Proper Shipping Name: RQ, DICHLOROMETHANE
Hazard Class: 6.1

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UN/NA: UN1593
Packing Group: III
Information reported for product/size: 1000L

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Methylene Chloride (75-09-2)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Methylene Chloride (75-09-2)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\--------

Ingredient	-SARA 302- RQ	TPQ	List	------SARA 313------ Chemical Catg.
Methylene Chloride (75-09-2)	No	No	Yes	No

--------\Federal, State & International Regulations - Part 2\--------

Ingredient	CERCLA	-RCRA- 261.33	TSCA- 8(d)
Methylene Chloride (75-09-2)	1000	U080	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: No Pressure: No
Reactivity: No (Pure / Liquid)

WARNING:
THIS PRODUCT CONTAINS A CHEMICAL(S) KNOWN TO THE STATE OF CALIFORNIA TO CAUSE CANCER.

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Australian Hazchem Code: 2Z
Poison Schedule: S5
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

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16. Other Information

NFPA Ratings: Health: **2** Flammability: **1** Reactivity: **0**
Label Hazard Warning:
WARNING! HARMFUL IF SWALLOWED, INHALED OR ABSORBED THROUGH SKIN. AFFECTS CENTRAL NERVOUS SYSTEM, LIVER, CARDIOVASCULAR SYSTEM, AND BLOOD. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. SUSPECT CANCER HAZARD. MAY CAUSE CANCER. Risk of cancer depends on level and duration of exposure.
Label Precautions:
Do not breathe vapor.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Keep away from heat and flame.
Do not get in eyes, on skin, or on clothing.
Label First Aid:
If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. In all cases, get medical attention.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 14.
Disclaimer:
****
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RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.

****

Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

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MSDS Number: **C2618** * * * * * *Effective Date:* ***08/10/04*** * * * * *Supercedes:* ***11/02/01***

MSDS Material Safety Data Sheet

From: **Mallinckrodt Baker, Inc.**
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS | J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

1-CHLOROBUTANE

1. Product Identification

Synonyms: Butylchloride; n-Butyl Chloride; n-propylcarbinyl chloride
CAS No.: 109-69-3
Molecular Weight: 92.58
Chemical Formula: CH3(CH2)3Cl
Product Codes:
J.T. Baker: E738, E740
Mallinckrodt: 1872

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Butyl Chloride	109-69-3	90 - 100%	Yes

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3. Hazards Identification

Emergency Overview

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. MAY BE HARMFUL IF SWALLOWED OR INHALED. MAY AFFECT CENTRAL NERVOUS SYSTEM. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 1 - Slight
Flammability Rating: 3 - Severe (Flammable)
Reactivity Rating: 2 - Moderate
Contact Rating: 2 - Moderate
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

Inhalation:
Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. High concentrations have a narcotic effect.
Ingestion:
Causes irritation to the gastrointestinal tract. Symptoms may include nausea, vomiting and diarrhea. Toxic! May cause systemic poisoning with symptoms paralleling those of inhalation.
Skin Contact:
Causes irritation to skin. Symptoms include redness, itching, and pain.
Eye Contact:
Causes irritation, redness, and pain.
Chronic Exposure:
Similar compounds demonstrate toxic effects after repeated exposures too low to result in acute effects.
Aggravation of Pre-existing Conditions:
No information found.

---

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult,

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give oxygen. Call a physician.

Ingestion:

Induce vomiting immediately as directed by medical personnel. Never give anything by mouth to an unconscious person. Call a physician.

Skin Contact:

In case of contact, immediately flush skin with plenty of soap and water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. Call a physician.

Eye Contact:

Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

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5. Fire Fighting Measures

Fire:

Flash point: -9C (16F) CC
Autoignition temperature: 240C (464F)
Flammable limits in air % by volume:
lel: 1.8; uel: 10.1
Extremely Flammable.

Explosion:

Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Contact with strong oxidizers may cause fire. Sealed containers may rupture when heated. Vapors can flow along surfaces to distant ignition source and flash back. Sensitive to static discharge.

Fire Extinguishing Media:

Dry chemical, foam or carbon dioxide. Water may be ineffective. Water spray may be used to keep fire exposed containers cool. Water may be used to flush spills away from exposures and to dilute spills to non-flammable mixtures.

Special Information:

In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Combustion by-products include phosgene and hydrogen chloride gases. This highly flammable liquid must be kept from sparks, open flame, hot surfaces, and all sources of heat and ignition.

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6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste

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container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures.

J. T. Baker SOLUSORB® solvent adsorbent is recommended for spills of this product.

---

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

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8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
None established.
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures as low as possible. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices,* most recent edition, for details.
Personal Respirators (NIOSH Approved):
For conditions of use where exposure to the substance is apparent and engineering controls are not feasble, consult an industrial hygienist. For emergencies, or instances where the exposure levels are not known, use a full-facepiece positive-pressure, air-supplied respirator. WARNING: Air purifying respirators do not protect workers in oxygen-deficient atmospheres.
Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.
Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

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9. Physical and Chemical Properties

Appearance:
Clear, colorless liquid.
Odor:
Unpleasant odor.
Solubility:
Practically insoluble in water.
Specific Gravity:
0.88 @ 25C/4C
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100
Boiling Point:
78C (172F)
Melting Point:
-123C (-189F)
Vapor Density (Air=1):
3.2
Vapor Pressure (mm Hg):
80 @ 20C (68F)
Evaporation Rate (BuAc=1):
No information found.

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10. Stability and Reactivity

Stability:
Stable at room temperature in sealed containers.
Hazardous Decomposition Products:
May produce carbon monoxide, carbon dioxide, hydrogen chloride and phosgene when heated to decomposition. Oxides of carbon as well as ionic or oxidized halogen.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Strong oxidizers.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

---

11. Toxicological Information

Oral rat LD50: 2670 mg/kg Irritation data: skin rabbit, Open Draize, 10mg/24H, mild. Eye rabbit, Standard Draize, 500mg/24-hour, mild. Investigated as a tumorigen and mutagen.

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  --------\Cancer Lists\----------------------------------------------
-------
                                          ---NTP Carcinogen---
  Ingredient                              Known    Anticipated    IARC
Category
  ----------------------------------   -----    -----------    ------
-------
  Butyl Chloride (109-69-3)               No          No
None
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12. Ecological Information

Environmental Fate:
When released into the soil, this material may leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released to water, this material is expected to quickly evaporate. When released into the water, this material is expected to have a half-life between 1 and 10 days. This material has an estimated bioconcentration factor (BCF) of less than 100. This material is not expected to significantly bioaccumulate. When released into the air, this material is expected to be readily degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material may be removed from the atmosphere to a moderate extent by wet deposition. When released into the air, this material is expected to have a half-life between 1 and 10 days.
Environmental Toxicity:
No information found.

---

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

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14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: CHLOROBUTANES
Hazard Class: 3
UN/NA: UN1127
Packing Group: II
Information reported for product/size: 4L

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International (Water, I.M.O.)

Proper Shipping Name: CHLOROBUTANES
Hazard Class: 3
UN/NA: UN1127
Packing Group: II
Information reported for product/size: 4L

International (Air, I.C.A.O.)

Proper Shipping Name: CHLOROBUTANES
Hazard Class: 3
UN/NA: UN1127
Packing Group: II
Information reported for product/size: 4L

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15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Butyl Chloride (109-69-3)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Butyl Chloride (109-69-3)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\--------

Ingredient	-SARA 302- RQ	TPQ	List	------SARA 313------ Chemical Catg.
Butyl Chloride (109-69-3)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\--------

Ingredient	CERCLA	-RCRA- 261.33	- TSCA- 8(d)
Butyl Chloride (109-69-3)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: No Fire: Yes Pressure: No
Reactivity: No (Pure / Liquid)

Australian Hazchem Code: 3[Y]E
Poison Schedule: None allocated.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **2** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. MAY BE HARMFUL IF SWALLOWED OR INHALED. MAY AFFECT CENTRAL NERVOUS SYSTEM. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT.
Label Precautions:
Keep away from heat, sparks and flame.
Keep container closed.
Avoid breathing vapor.
Use only with adequate ventilation.
Avoid contact with eyes, skin and clothing.
Wash thoroughly after handling.
Label First Aid:
If swallowed, induce vomiting immediately as directed by medical personnel. Never give anything by mouth to an unconscious person. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In all cases call a physician.
Product Use:
Laboratory Reagent.
Revision Information:
No Changes.
Disclaimer:
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Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

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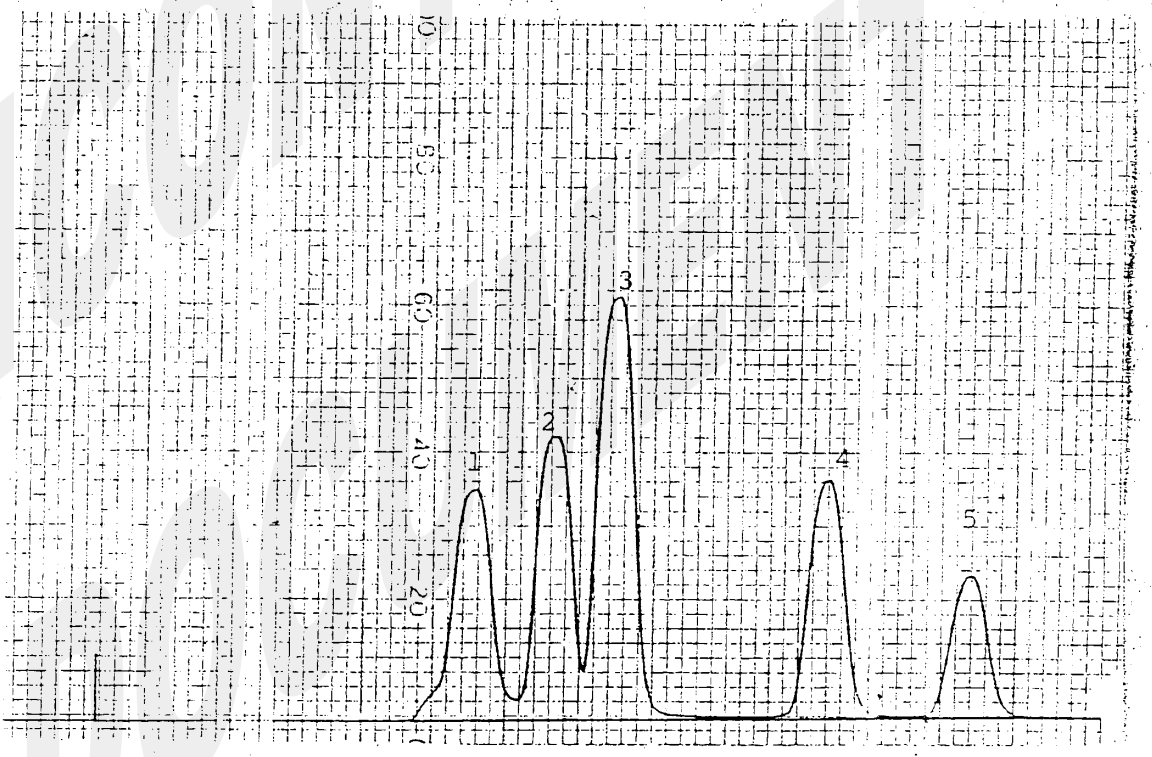
CALIBRATION SOLUTION CHROMATOGRAM



1 - Corn Oil
2 - Bis(2-ethylhexyl) phthalate
3 - Methoxychlor
4 - Perylene
5 - Sulfur

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Details of method PCB/Pest Cleanup5

6/27/02 3:28:08 PM

Method Description: PCB/Pest Cleanup 5□□Made10/08/01 ST

Method is Cleanup type
Dump Time1: 13.00 min
Collect Time1: 9.70 min
Dump Time2: 0.00 min
Collect Time2: 0.00 min
Wash Time: 10.00 min

Pump Rate: 5.00 ml/min
No of Rinses: 1
Time needle is rinsed: 10 sec
Column Type: EnviroSEP-ABC

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Heading 1: CPC CLEANUP #16
Heading 2: BATCH 1482 BNA
Heading 3: TM
Initial Vol: 2.00000
Run: Thu Jan 01 00:00:00 1970 Entered: Mon Mar 04 15:51:27 2002

Position	Sample ID	Initial Wt	CAN.DISP	VTX LOSS	FIL ASP	FIL DISP	HPLC.NUM	HPLC.ASP
1	MPB 1482	0.0000	9.8066	-0.0262	-12.0268	11.2868	1.0000	-8.6170
2	LCS 1482	0.0000	9.7982	-0.0348	-11.9856	11.2830	2.0000	-8.4240
3	MS 1482	0.0000	9.6185	-0.0165	-12.0198	11.4887	3.0000	-8.3868
4	MSD 1482	0.0000	9.5806	-0.0263	-11.6361	11.0680	4.0000	-8.4751
5	02 128 01	0.0000	9.6051	-0.0354	-11.9338	11.2939	5.0000	-8.3738
6	02 128 02	0.0000	9.5695	-0.0219	-11.8437	11.5758	6.0000	-8.3351
7	02 128 03	0.0000	9.5293	-0.0280	-11.4772	11.0126	7.0000	-8.3380
8	02 128 04	0.0000	9.5569	-0.0307	-11.8237	11.3518	8.0000	-8.4410
9	02 128 05	0.0000	9.5106	-0.0232	-11.9625	11.3966	9.0000	-8.3637
10	02 128 06	0.0000	9.6094	-0.0215	-11.9701	11.4254	10.0000	-8.3801
11	02 128 07	0.0000	9.5879	-0.0378	-11.4869	10.9140	11.0000	-8.4851
12	02 128 08	0.0000	9.6299	-0.0211	-11.9220	11.0937	12.0000	-8.3106
13	02 128 09	0.0000	9.6021	-0.0307	-11.8795	11.1316	13.0000	-8.3125
14	02 128 10	0.0000	9.4512	-0.0244	-11.6654	10.9399	14.0000	-8.4418
15	02 128 11	0.0000	9.5090	-0.0782	-11.2849	10.7734	15.0000	-8.3351
16	02 128 12	0.0000	9.5022	-0.0219	-11.6725	11.0919	16.0000	-8.3389
17	RINSE	0.0000	9.4821	-0.0255	-12.1338	11.6314	17.0000	-8.4527
18	RINSE	0.0000	9.4093	-0.0194	-11.5657	11.5300	18.0000	-8.4568

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Date	Start Time	Inject Time	End Time	Results	Method	Vial	Sample ID	Final Volume
6/24/02	2:24:37 PM	2:26:18 PM	2:59:09 PM	Completed run	PCB/Pest Cleanup5	1	Rinse #1	
6/24/02	2:59:09 PM	3:00:50 PM	3:33:41 PM	Completed run	PCB/Pest Cleanup5	2	MPB 4954	
6/24/02	3:33:41 PM	3:35:22 PM	4:08:13 PM	Completed run	PCB/Pest Cleanup5	3	LCS 4954	
6/24/02	4:08:13 PM	4:09:54 PM	4:42:45 PM	Completed run	PCB/Pest Cleanup5	4	MS (193-01)	
6/24/02	4:42:45 PM	4:44:26 PM	5:17:17 PM	Completed run	PCB/Pest Cleanup5	5	MSD (193-01)	
6/24/02	5:17:17 PM	5:18:58 PM	5:51:49 PM	Completed run	PCB/Pest Cleanup5	6	0206176-01	
6/24/02	5:51:49 PM	5:53:31 PM	6:26:22 PM	Completed run	PCB/Pest Cleanup5	7	0206188-01	
6/24/02	6:26:22 PM	6:28:03 PM	7:00:54 PM	Completed run	PCB/Pest Cleanup5	8	0206188-02	
6/24/02	7:00:54 PM	7:02:36 PM	7:35:27 PM	Completed run	PCB/Pest Cleanup5	9	0206188-03	
6/24/02	7:35:27 PM	7:37:09 PM	8:09:59 PM	Completed run	PCB/Pest Cleanup5	10	0206188-04	
6/24/02	8:09:59 PM	8:11:42 PM	8:44:32 PM	Completed run	PCB/Pest Cleanup5	11	0206188-05	
6/24/02	8:44:32 PM	8:46:15 PM	9:19:05 PM	Completed run	PCB/Pest Cleanup5	12	0206188-06	
6/24/02	9:19:05 PM	9:20:48 PM	9:53:38 PM	Completed run	PCB/Pest Cleanup5	13	0206188-07	
6/24/02	9:53:38 PM	9:55:21 PM	10:28:11 PM	Completed run	PCB/Pest Cleanup5	14	0206193-01	
6/24/02	10:28:12 PM	10:29:53 PM	11:02:44 PM	Completed run	PCB/Pest Cleanup5	15	0206193-02	
6/24/02	11:02:44 PM	11:04:26 PM	11:37:17 PM	Completed run	PCB/Pest Cleanup5	16	0206195-01	
6/24/02	11:37:17 PM	11:38:59 PM	12:11:49 AM	Completed run	PCB/Pest Cleanup5	17	0206195-02	
6/25/02	12:11:49 AM	12:13:31 AM	12:46:22 AM	Completed run	PCB/Pest Cleanup5	18	0206195-04	
6/25/02	12:46:22 AM	12:48:03 AM	1:20:54 AM	Completed run	PCB/Pest Cleanup5	19	0206195-05	
6/25/02	1:20:54 AM	1:22:35 AM	1:55:26 AM	Completed run	PCB/Pest Cleanup5	20	0206222-01	
6/25/02	1:55:26 AM	1:57:07 AM	2:29:58 AM	Completed run	PCB/Pest Cleanup5	21	Rinse #2	

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O-I CORPORATION

DEQ Environmental Lab
Lansing, MI 48906
Phone: 517-335-9800
Fax: 517-335-9600
WWW:

	Name	Date
Approval 1:		
Approval 2:		

Sequence:	4954	Start of Run:	6/24/02 2:24:37PM
Operator:	DEQDWRP	Overall Results:	Completed run

Configuration Details:

Syringe Size(ml):	10.00	Detector Type:	ISCO UA-6	Sample Tray:	60 16 * 100 mm tubes
Loop Size (ml):	5.00	Using UV Detector:	Yes	Collect Tray:	TurboVap 125 mL tubes
Flow Rate(ml/min):	5.00	Plotting UV Data:	No	Column Type:	EnviroSEP-ABC
Bypass Valve:	Yes			Solvent Type:	Methylene chloride

Sample Details:

Sample ID: 0206176-01

Sample Vial:	6	Sample Type:	Sample		
First Collect Vial:	6	Sample Vol (ml)	10.00		
Last Collect Vial:	6	Sample Wt/Vol:		Priority:	Normal
Method:	PCB/Pest Cleanup5				
Comments:					

Sample ID: 0206188-01

Sample Vial:	7	Sample Type:	Sample		
First Collect Vial:	7	Sample Vol (ml)	10.00		
Last Collect Vial:	7	Sample Wt/Vol:		Priority:	Normal
Method:	PCB/Pest Cleanup5				
Comments:					

Sample ID: 0206188-02

Sample Vial:	8	Sample Type:	Sample		
First Collect Vial:	8	Sample Vol (ml)	10.00		
Last Collect Vial:	8	Sample Wt/Vol:		Priority:	Normal
Method:	PCB/Pest Cleanup5				
Comments:					

Sample ID: 0206188-03

Sample Vial:	9	Sample Type:	Sample		
First Collect Vial:	9	Sample Vol (ml)	10.00		
Last Collect Vial:	9	Sample Wt/Vol:		Priority:	Normal
Method:	PCB/Pest Cleanup5				
Comments:					

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Sequence: 4954 Start of Run: 6/24/02 2:24:37PM
Operator: DEQDWRP Overall Results: Completed run

Sample ID: 0206188-04

Sample Vial: 10 Sample Type: Sample
First Collect Vial: 10 Sample Vol (ml) 10.00
Last Collect Vial: 10 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: 0206188-05

Sample Vial: 11 Sample Type: Sample
First Collect Vial: 11 Sample Vol (ml) 10.00
Last Collect Vial: 11 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: 0206188-06

Sample Vial: 12 Sample Type: Sample
First Collect Vial: 12 Sample Vol (ml) 10.00
Last Collect Vial: 12 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: 0206188-07

Sample Vial: 13 Sample Type: Sample
First Collect Vial: 13 Sample Vol (ml) 10.00
Last Collect Vial: 13 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: 0206193-01

Sample Vial: 14 Sample Type: Sample
First Collect Vial: 14 Sample Vol (ml) 10.00
Last Collect Vial: 14 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: 0206193-02

Sample Vial: 15 Sample Type: Sample
First Collect Vial: 15 Sample Vol (ml) 10.00
Last Collect Vial: 15 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: 0206195-01

Sample Vial: 16 Sample Type: Sample
First Collect Vial: 16 Sample Vol (ml) 10.00
Last Collect Vial: 16 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

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Sequence: 4954 Start of Run: 6/24/02 2:24:37PM
Operator: DEQDWRP Overall Results: Completed run

Sample ID: 0206195-02

Sample Vial: 17 Sample Type: Sample
First Collect Vial: 17 Sample Vol (ml) 10.00
Last Collect Vial: 17 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: 0206195-04

Sample Vial: 18 Sample Type: Sample
First Collect Vial: 18 Sample Vol (ml) 10.00
Last Collect Vial: 18 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: 0206195-05

Sample Vial: 19 Sample Type: Sample
First Collect Vial: 19 Sample Vol (ml) 10.00
Last Collect Vial: 19 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: 0206222-01

Sample Vial: 20 Sample Type: Sample
First Collect Vial: 20 Sample Vol (ml) 10.00
Last Collect Vial: 20 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: LCS 4954

Sample Vial: 3 Sample Type: Sample
First Collect Vial: 3 Sample Vol (ml) 10.00
Last Collect Vial: 3 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: MPB 4954

Sample Vial: 2 Sample Type: Sample
First Collect Vial: 2 Sample Vol (ml) 10.00
Last Collect Vial: 2 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: MS (193-01)

Sample Vial: 4 Sample Type: Sample
First Collect Vial: 4 Sample Vol (ml) 10.00
Last Collect Vial: 4 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

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Sequence: 4954 Start of Run: 6/24/02 2:24:37PM
Operator: DEQDWRP Overall Results: Completed run

Sample ID: MSD (193-01)

Sample Vial: 5 Sample Type: Sample
First Collect Vial: 5 Sample Vol (ml) 10.00
Last Collect Vial: 5 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: Rinse #1

Sample Vial: 1 Sample Type: Sample
First Collect Vial: 1 Sample Vol (ml) 10.00
Last Collect Vial: 1 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sample ID: Rinse #2

Sample Vial: 21 Sample Type: Sample
First Collect Vial: 21 Sample Vol (ml) 10.00
Last Collect Vial: 21 Sample Wt/Vol: Priority: Normal
Method: PCB/Pest Cleanup5
Comments:

Sequence Summary:

Calibrations and Check Standards Processed: 0
Total Errors Encountered: 0
Total Run Time: 0 Day(s), 12 Hour(s), 5 Minute(s), 21 Second(s)

Method Details:

Name: PCB/Pest Cleanup5

Dump Time 1 (min): 13.00 Quantity of Rinses: 1 Creator: DEQDWRP
Dump Time 2 (min): 0.00 Needle Rinse Time (sec): 10 Last Edited: 10/8/01 11:46:23AM
Collect Time 1 (min): 9.70 Revision: 1
Collect Time 2 (min): 0.00 Rinse Time (min): 10.00 Type: Cleanup
Comments: PCB/Pest Cleanup 5
Made10/08/01 ST

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Sequence: 4954 Start of Run: 6/24/02 2:24:37PM

Operator: DEQDWRP Overall Results: Completed run

Result Details

Start Time	Completion Time	Results	Sample Vial No.	First Collect Vial	Last Collect V. Number
6/24/02 2:24:37PM	6/24/02 2:59:09PM	Completed run	1	1	
6/24/02 2:59:09PM	6/24/02 3:33:41PM	Completed run	2	2	2
6/24/02 3:33:41PM	6/24/02 4:08:13PM	Completed run	3	3	3
6/24/02 4:08:13PM	6/24/02 4:42:45PM	Completed run	4	4	4
6/24/02 4:42:45PM	6/24/02 5:17:17PM	Completed run	5	5	5
6/24/02 5:17:17PM	6/24/02 5:51:49PM	Completed run	6	6	6
6/24/02 5:51:49PM	6/24/02 6:26:22PM	Completed run	7	7	7
6/24/02 6:26:22PM	6/24/02 7:00:54PM	Completed run	8	8	8
6/24/02 7:00:54PM	6/24/02 7:35:27PM	Completed run	9	9	9
6/24/02 7:35:27PM	6/24/02 8:09:59PM	Completed run	10	10	10
6/24/02 8:09:59PM	6/24/02 8:44:32PM	Completed run	11	11	11
6/24/02 8:44:32PM	6/24/02 9:19:05PM	Completed run	12	12	12
6/24/02 9:19:05PM	6/24/02 9:53:38PM	Completed run	13	13	13
6/24/02 9:53:38PM	6/24/02 10:28:11PM	Completed run	14	14	14
6/24/02 10:28:12PM	6/24/02 11:02:44PM	Completed run	15	15	15
6/24/02 11:02:44PM	6/24/02 11:37:17PM	Completed run	16	16	16
6/24/02 11:37:17PM	6/25/02 12:11:49AM	Completed run	17	17	17
6/25/02 12:11:49AM	6/25/02 12:46:22AM	Completed run	18	18	18
6/25/02 12:46:22AM	6/25/02 1:20:54AM	Completed run	19	19	19
6/25/02 1:20:54AM	6/25/02 1:55:26AM	Completed run	20	20	20
6/25/02 1:55:26AM	6/25/02 2:29:58AM	Completed run	21	21	21

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Occasionally, samples will be too thick to be filtered through the benchmate. These samples need to be hand filtered using the following procedure:

Soil samples are extracted according to internal procedure SOP 401 and brought to 10 mL in MeCl.

- Attach an Acrodisc filter (Section 9.20) to a syringe (Section 9.21).
- Pour the sample extract into the syringe. Filter into a 10 mL pre-marked culture tube (Section 9.1).
- Transfer the label to the new tube.

Cover the tube with foil. Cover with a star cap to hold the fill in place (Section 9.2). Samples are run on either benchmate #14 or benchmate #16 with the 10 mL start procedure.

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Zymark BenchMate 2.5

Zymark BenchMate Procedure : BM #14 10 ML START GPC CLEAN-UP

Step 1 : Wash syringe with 10 ml of MeCl2
Step 2 : Wash LC injector with 10 ml of MeCl2
Step 3 : Vortex for 10 seconds at speed 2
Step 4 : inject sample on LC 1 with run time of 9 minutes
Step 5 : Wash syringe with 10 ml of MeCl2
Step 6 : Wash LC injector with 10 ml of MeCl2
Step 7 : Pause for 18 minutes
Step 8 : Wash syringe with 10 ml of MeCl2
Step 9 : Wash syringe with 10 ml of MeCl2
Step 10 : Wash LC injector with 10 ml of MeCl2
Step 11 : Wash LC injector with 10 ml of MeCl2
Step 12 : END

SETUP PARAMETERS - 1

SAMPLE TUBE
Initial Volume: 10.0 ml

FLOW RATES
Aspirate: 0.50 ml/sec
Dispense: 1.00 ml/sec
Internal Std: 0.12 ml/sec
Mix: 1.50 ml/sec
Filter: 0.10 ml/sec
Air Push: 0.15 ml/sec

DISPENSING
Liquid driven: Y Y=YES N=NO

AUTOWASH PARAMETERS
Reagent Vol: 0.00 ml
Sample Vol: 0000 ml

GRAVIMETRIC PARAMETERS
Gravimetric on: Y Y=YES N=NO
Tolerance: 20%
Tare Sample Tubes: N Y=YES N=NO

RACK PARAMETERS
LV Tube Numbering: Y Y=YES N=NO
Reset to Sample 1: N Y=YES N=NO

SETUP PARAMETERS - 2

SPE PARAMETERS
Cond Flow: 0.25 ml/sec
Load Flow: 0.15 ml/sec
Rinse Flow: 0.25 ml/sec
Elute Flow: 0.15 ml/sec
Push Delay: 5 sec
Air Factor: 1.0
Save Columns: N Y=YES N=NO

LC PARAMETERS
Inject Load Vol: 7.00 ml
Calibrate every: 0 samples
Calib Replicates: 1
Calibration Reagent: 1
Calib Wash Vol: 0.00 ml

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Zymark BenchMate Procedure : BM #14 10 ML START GPC CLEAN-UP

NAME REAGENTS

Reagent 1 : MeCl2 ; Density : 1.3170
Reagent 2 : REAGENT 2 ; Density : 1.0000
Reagent 3 : REAGENT 3 ; Density : 1.0000
Reagent 4 : REAGENT 4 ; Density : 1.0000
Reagent 5 : REAGENT 5 ; Density : 1.0000
Reagent 6 : REAGENT 6 ; Density : 1.0000

NAME INTERNAL STANDARD

Standard : INTERNAL STD ; Density : 1.0000

BenchMate Table Setup

THE ESTIMATED TIME FOR ONE SAMPLE IS: 40.2 MINUTES.

NO FILTERS ARE USED.
NO SPE COLUMNS ARE USED.

Tubes will be processed starting at Rack 1 position 1,
through all Racks until an empty position is encountered.

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Zymark BenchMate 3.10

Zymark BenchMate Procedure : BM II 10 ML START GPC CLEAN-UP

Step 1 : Wash syringe with 10 ml of MeCl2
Step 2 : Wash LC injector with 10 ml of MeCl2
Step 3 : Vortex for 10 seconds at speed 2
Step 4 : inject sample on LC with run time of 9 minutes
Step 5 : Wash LC injector with 10 ml of MeCl2
Step 6 : Wash syringe with 10 ml of MeCl2
Step 7 : Pause for 18 minutes
Step 8 : Wash syringe with 10 ml of MeCl2
Step 9 : Wash syringe with 10 ml of MeCl2
Step 10 : Wash LC injector with 10 ml of MeCl2
Step 11 : Wash LC injector with 10 ml of MeCl2
Step 12 : END

SETUP PARAMETERS

SAMPLE TUBE
Initial Volume: 10.0 ml

GRAVIMETRIC PARAMETERS
Gravimetric on: Y Y=YES N=NO
Tare Sample Tubes: N Y=YES N=NO

FLOW RATES
Aspirate: 0.50 ml/sec
Dispense: 1.00 ml/sec
Internal Std: 0.12 ml/sec
Mix: 1.50 ml/sec
Air Push: 0.15 ml/sec

RACK PARAMETERS
LV Tube Numbering: Y Y=YES N=NO
Reset to Sample 1: N Y=YES N=NO

FILTER PARAMETERS
Filter Flow Rate: 0.21 ml/sec
Filtrate Confirm: N Y=YES N=NO
Filtrate Density: 1.00 g/ml
Minimum Amount: 0%

DISPENSING
Liquid driven: Y Y=YES N=NO

AUTOWASH PARAMETERS
Rinse Reagent: 1
Reagent Vol: 0.00 ml
Sample Vol: 0.00 ml

SETUP SPE PARAMETERS

SPE PARAMETERS
Cond Flow: 0.25 ml/sec
Aspirate Flow: 0.25 ml/sec
Load Flow: 0.15 ml/sec
Rinse Flow: 0.25 ml/sec
Elute Flow: 0.15 ml/sec
Push Delay: 5 sec
Air Factor: 1.0
Save Columns: N Y=YES N=NO
Save Samples: N Y=YES N=NO

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SETUP LC PARAMETERS

LC PARAMETERS
Inject Sip Vol: 7.00 ml
Inject Delay Time: 99 sec
Loop in Load Pos: N Y=YES N=NO
Inject Loop Time: 99 sec

ENCORE PARAMETERS
Load disk methods: Y Y=YES N=NO
Lock pump keypad: Y Y=YES N=NO
Initialization Time: 0 min
Equilibration Time: 0 min
Shutdown method: 0

LC CALIBRATION STANDARDS
Calibrate every: 0 samples
INJECT START DURING END
LC Std 1: 0 0 0
LC Std 2: 0 0 0
LC Std 3: 0 0 0
LC Std 4: 0 0 0
LC Std 5: 0 0 0
LC Std 6: 0 0 0
Calib Wash Vol: 0.00 ml
Calib Wash Reagent: 1

SETUP EasyFill PARAMETERS

EasyFill PARAMETERS
EasyFill vial type: 2
Sense vials: Y Y=YES N=NO
External needle wash: Y Y=YES N=NO

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Zymark BenchMate Procedure : BM II 10 ML START GPC CLEAN-UP

NAME REAGENTS

Reagent 1 : MeCl2 ; Density : 1.3170
Reagent 2 : REAGENT 2 ; Density : 1.0000
Reagent 3 : REAGENT 3 ; Density : 1.0000
Reagent 4 : REAGENT 4 ; Density : 1.0000
Reagent 5 : REAGENT 5 ; Density : 1.0000
Reagent 6 : REAGENT 6 ; Density : 1.0000

NAME INTERNAL STANDARD

Standard : INTERNAL STD ; Density : 1.0000

BenchMate Table Setup

THE ESTIMATED TIME FOR ONE SAMPLE IS: 34.1 MINUTES.

NO FILTERS ARE USED.
NO SPE COLUMNS ARE USED.

Tubes will be processed starting at Rack 1 position 1,
through all Racks until an empty position is encountered.

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OI SAMPLE ID EXAMPLE

Sample	Sample ID	Method	Sample Ty	Comments	First Collect	Last Collect
1	Rinse #1	PCB/Pest Cleanup	Sample		1	1
2	MPB 4954	PCB/Pest Cleanup	Sample		2	2
3	LCS 4954	PCB/Pest Cleanup	Sample		3	3
4	MS (193-01)	PCB/Pest Cleanup	Sample		4	4
5	MSD (193-01)	PCB/Pest Cleanup	Sample		5	5
6	0206176-01	PCB/Pest Cleanup	Sample		6	6
7	0206188-01	PCB/Pest Cleanup	Sample		7	7
8	0206188-02	PCB/Pest Cleanup	Sample		8	8
9	0206188-03	PCB/Pest Cleanup	Sample		9	9
10	0206188-04	PCB/Pest Cleanup	Sample		10	10
11	0206188-05	PCB/Pest Cleanup	Sample		11	11
12	0206188-06	PCB/Pest Cleanup	Sample		12	12
13	0206188-07	PCB/Pest Cleanup	Sample		13	13
14	0206193-01	PCB/Pest Cleanup	Sample		14	14
15	0206193-02	PCB/Pest Cleanup	Sample		15	15
16	0206195-01	PCB/Pest Cleanup	Sample		16	16
17	0206195-02	PCB/Pest Cleanup	Sample		17	17
18	0206195-04	PCB/Pest Cleanup	Sample		18	18
19	0206195-05	PCB/Pest Cleanup	Sample		19	19
20	0206222-01	PCB/Pest Cleanup	Sample		20	20
21	Rinse #2	PCB/Pest Cleanup	Sample		21	21

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OI CALIBRATION SEQUENCE METHOD

Sample	Sample ID	Method	Sample Ty	Comments	First Collect	Last Collect
1	Rinse	calib	Calibrati		1	1
2	Cal	calib	Calibrati		1	1
3	Rinse	calib	Calibrati		1	1

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Details of method calib

6/27/02 3:59:29 PM

Method Description: 5 ml/min calib

Method is Cleanup type
Dump Time1: 30.00 min
Collect Time1: 0.00 min
Dump Time2: 0.00 min
Collect Time2: 0.00 min
Wash Time: 10.00 min

Pump Rate: 5.00 ml/min
No of Rinses: 1
Time needle is rinsed: 10 sec
Column Type: EnviroSEP-ABC

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Configuration settings for sequence Calibration

3/11/02 8:14:23 AM

Pump Rate:	5.00 ml/min
Loop Size:	5.00 ml
Syringe Size:	10 ml
Column Type:	EnviroSEP-ABC
Detector Type:	ISCO UA-6
Number of Detectors:	1
Sample Tray:	60 16 * 100 mm tubes
Collect Tray:	TurboVap 125 mL tubes
Plotting Detector Data:	NO
Bypass Valve installed:	YES

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Method Data Page

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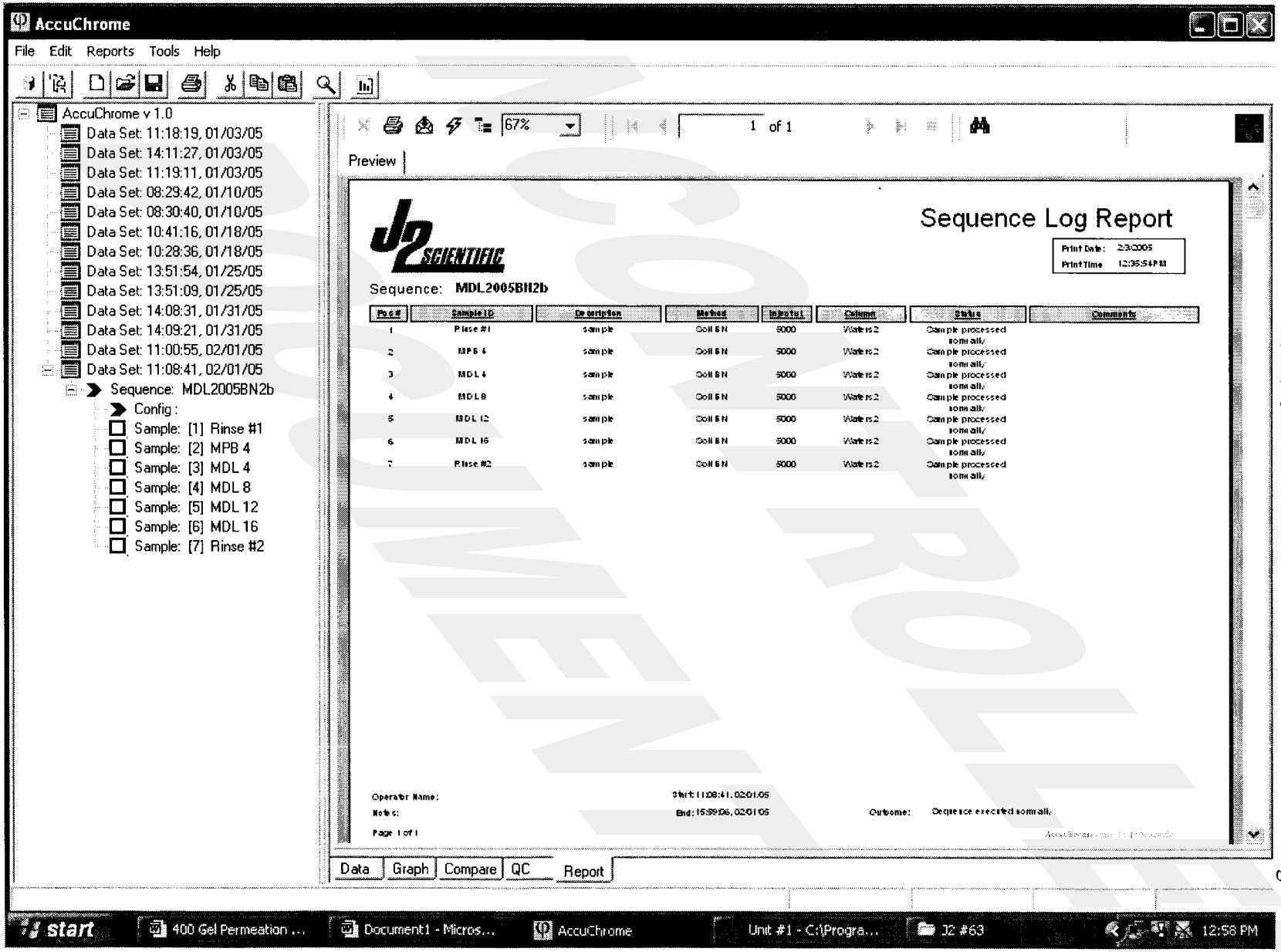
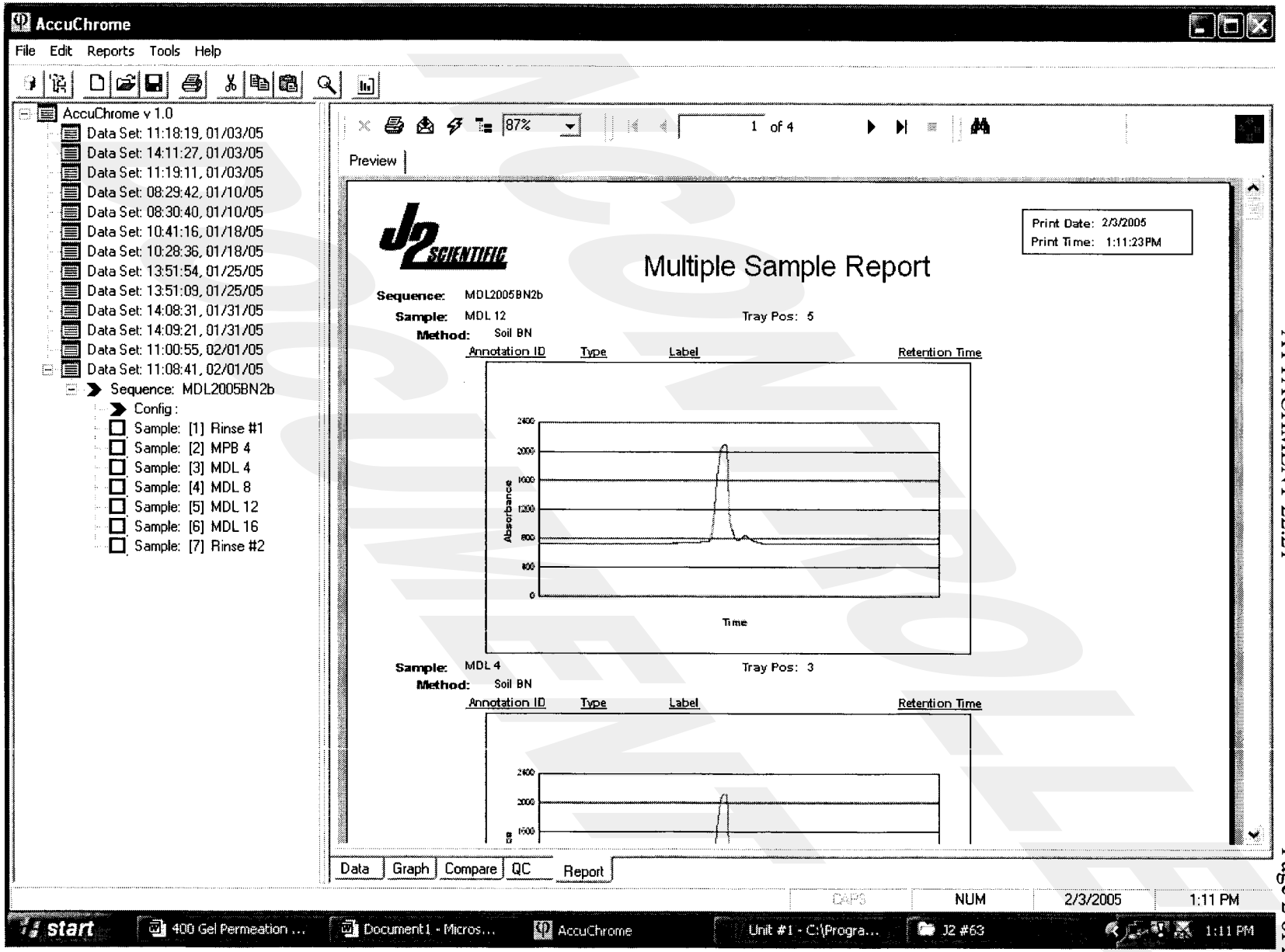
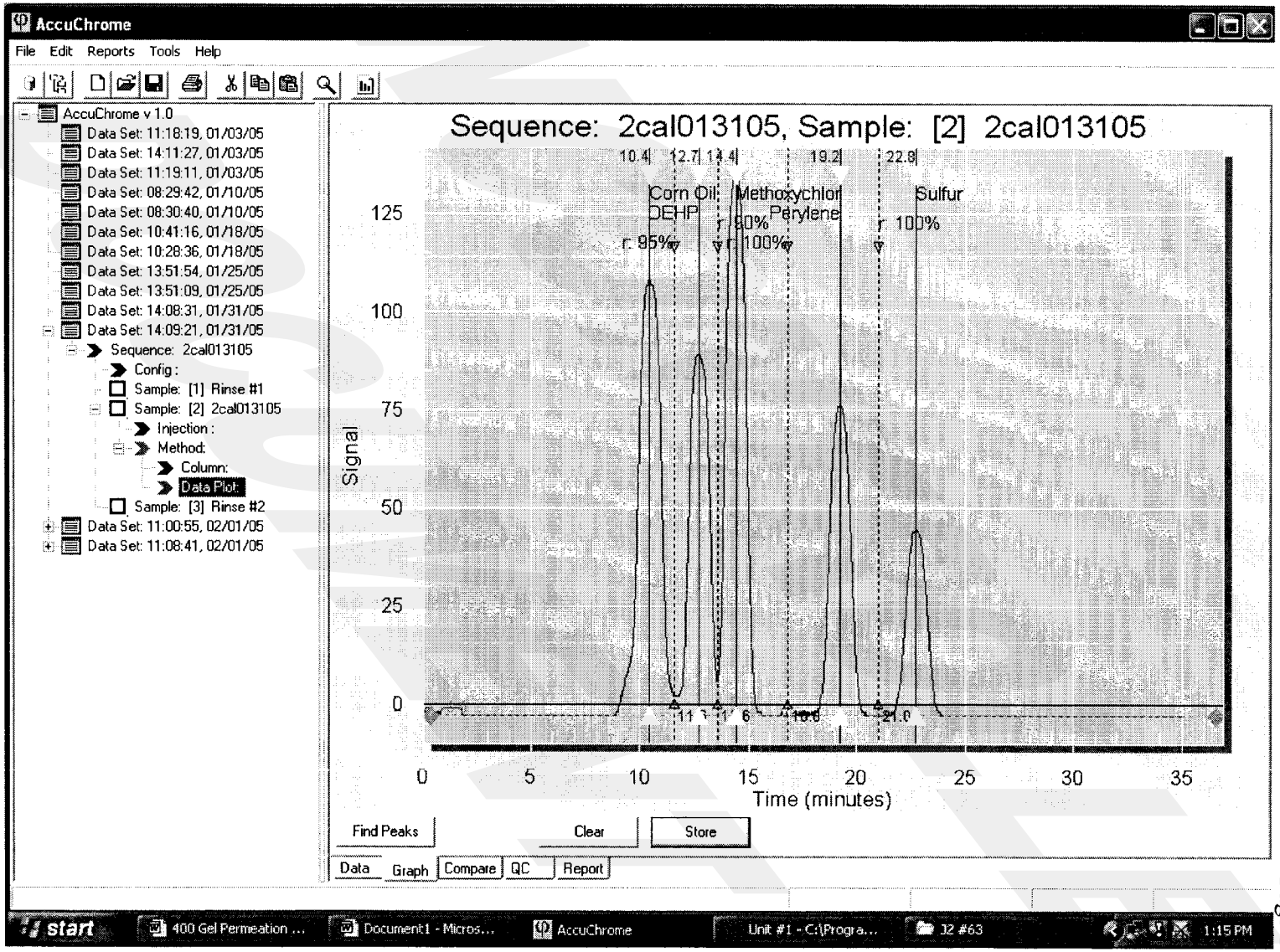


DOCUMENT CONTROL


AccuChrome
File Edit Reports Tools Help
AccuChrome v 1.0
Data Set: 11:18:19, 01/03/05
Data Set: 14:11:27, 01/03/05
Data Set: 11:19:11, 01/03/05
Data Set: 08:29:42, 01/10/05
Data Set: 08:30:40, 01/10/05
Data Set: 10:41:16, 01/18/05
Data Set: 10:28:36, 01/18/05
Data Set: 13:51:54, 01/25/05
Data Set: 13:51:09, 01/25/05
Data Set: 14:08:31, 01/31/05
Data Set: 14:09:21, 01/31/05
Data Set: 11:00:55, 02/01/05
Data Set: 11:08:41, 02/01/05
Sequence: MDL2005BN2b
Config :
Sample: [1] Rinse #1
Sample: [2] MPB 4
Sample: [3] MDL 4
Sample: [4] MDL 8
Sample: [5] MDL 12
Sample: [6] MDL 16
Sample: [7] Rinse #2
87%
1 of 4
Preview
Print Date: 2/3/2005
Print Time: 1:11:23PM
Multiple Sample Report
Sequence: MDL2005BN2b
Sample: MDL 12
Tray Pos: 5
Method: Soil BN
Annotation ID
Type
Label
Retention Time
Absorbance
Time
Sample: MDL 4
Tray Pos: 3
Method: Soil BN
Annotation ID
Type
Label
Retention Time
Data
Graph
Compare
QC
Report
CAPS
NUM
2/3/2005
1:11 PM
start
400 Gel Permeation ...
Document1 - Micros...
AccuChrome
Unit #1 - C:\Progra...
J2 #63
1:11 PM



ATTACHMENT 22.22

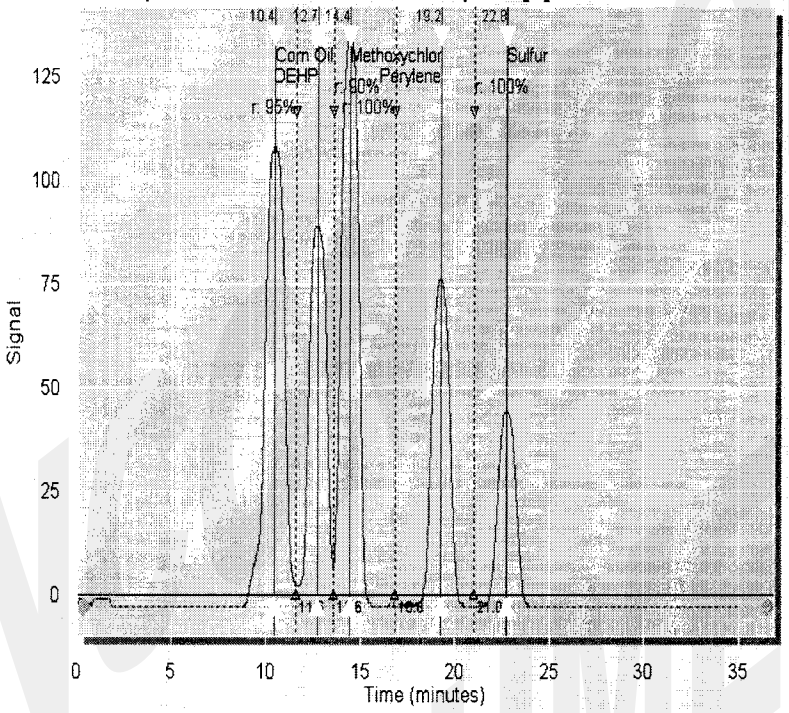
Print Date:
Print Time:

Column Calibration Report

Sample ID: 2cal013105
Sequence: 2cal013105
Description: sample
Volume: 5000
Method: calibration
Position: 2
Outcome: Sample processed normally

Start Time: 14:50:03, 01/31/05
End Time: 15:27:20, 01/31/05
Column Name: Waters2
Description: Waters 02-092204
Serial #: 1051J, 2171J, 1681L
Pressure Limit: 350

Sequence: 2cal013105, Sample: [2] 2cal013105



2cal013105

Peak	Sample	Retention Time	Pk Ht	Base	Res.	Tol.	P/F
1	Corn Oil	10.5	107	-3.0			
2	DEHP	12.7	89	-3.0	94.6	85.0	P
3	Methoxychlor	14.4	134	-3.0	90.2	85.0	P
4	Perylene	19.2	76	-3.0	100.0	85.0	P
5	Sulfur	22.8	45	-3.0	100.0	90.0	P

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Attachment 22.23

The lancer dishwasher has two programs that we use. Program 5 uses deionized water and program 4 uses tap water.

Functions	Possibility	Our Program
PREWASH 1 PERIOD	0-30 MINUTES	1 MINUTE
FILLING	HOT/COLD	HOT
PREWASH 1 TEMPERATURE	0-95°C	50°C
PREWASH 1 DETERGENT	0-6 MINUTES	24 SECONDS
PREWASH 1 ADDITIVE 2 (ACID)	0-6 MINUTES	0
PREWASH 2 PERIOD	0-30 MINUTES	2 MINUTES
FILLING	HOT/COLD	HOT
PREWASH 2 TEMPERATURE	0-95°C	65°C
PREWASH 2 DETERGENT	0-6 MINUTES	24 SECONDS
PREWASH 2 ADDITIVE 2	0-6 MINUTES	0
PREWASH 3 PERIOD	0-30 MINUTES	0
FILLING	HOT/COLD	0
PREWASH 3 TEMPERATURE	0-95°C	0
PREWASH 3 DETERGENT	0-6 MINUTES	0
PREWASH 3 ADDITIVE 2	0-6 MINUTES	0
WASH PERIOD	0-30 MINUTES	4 MINUTES
FILLING	HOT/COLD	HOT
WASH TEMPERATURE	0-95°C	80°C
WASH DETERGENT	0-6 MINUTES	1 MINUTE 6 SECONDS
WASH ADDITIVE 2	0-6 MINUTES	0
RUNNING WATER RINSE 1	0-9 MINUTES	2 MINUTES
ACID RINSE PERIOD	0-30 MINUTES	2 MINUTES
ACID	0-6 MINUTES	2 MINUTES
RUNNING WATER RINSE	0-9 MINUTES	7 MINUTES
COLD DEMI. WATER RINSE 1	0-30 MINUTES	0
COLD DEMI. WATER RINSE 2	0-30 MINUTES	0
COLD DEMI. WATER RINSE 3	0-30 MINUTES	0
COLD DEMI. WATER RINSE 4	0-3 MINUTES	0
HOT DEMI. WATER RINSE	0-30 MINUTES	2 MINUTES
HOT DEMI. TEMPERATURE	0-95°C	75°C
HOT DEMI. ADDITIVE	0-6 MINUTES	0
DRYING	0-90 MINUTES	25 MINUTES

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MSDS Number: **A0446** * * * * * *Effective Date:* ***05/20/04*** * * * * * *Supercedes:* ***02/12/04***

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

ACETONE

1. Product Identification

Synonyms: Dimethylketone; 2-propanone; dimethylketal
CAS No.: 67-64-1
Molecular Weight: 58.08
Chemical Formula: (CH3)2CO
Product Codes:
J.T. Baker: 5008, 5018, 5356, 5580, 9001, 9002, 9003, 9004, 9005, 9006, 9007, 9008, 9009, 9010, 9015, 9024, 9036, 9125, 9254, 9271, A134, V655
Mallinckrodt: 0018, 2432, 2435, 2437, 2438, 2440, 2443, 2445, 2850, H451, H580, H981

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Acetone	67-64-1	99 - 100%	Yes

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3. Hazards Identification

Emergency Overview

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL IF SWALLOWED OR INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM.

SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 2 - Moderate
Flammability Rating: 3 - Severe (Flammable)
Reactivity Rating: 0 - None
Contact Rating: 3 - Severe
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

Inhalation:
Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache. Higher concentrations can produce central nervous system depression, narcosis, and unconsciousness.

Ingestion:
Swallowing small amounts is not likely to produce harmful effects. Ingestion of larger amounts may produce abdominal pain, nausea and vomiting. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms are expected to parallel inhalation.

Skin Contact:
Irritating due to defatting action on skin. Causes redness, pain, drying and cracking of the skin.

Eye Contact:
Vapors are irritating to the eyes. Splashes may cause severe irritation, with stinging, tearing, redness and pain.

Chronic Exposure:
Prolonged or repeated skin contact may produce severe irritation or dermatitis.

Aggravation of Pre-existing Conditions:
Use of alcoholic beverages enhances toxic effects. Exposure may increase the toxic potential of chlorinated hydrocarbons, such as chloroform, trichloroethane.

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4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.
Ingestion:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately.
Skin Contact:
Immediately flush skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting upper and lower eyelids occasionally. Get medical attention.

---

5. Fire Fighting Measures

Fire:
Flash point: -20C (-4F) CC
Autoignition temperature: 465C (869F)
Flammable limits in air % by volume:
lel: 2.5; uel: 12.8
Extremely Flammable Liquid and Vapor! Vapor may cause flash fire.
Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Vapors can flow along surfaces to distant ignition source and flash back. Contact with strong oxidizers may cause fire. Sealed containers may rupture when heated. This material may produce a floating fire hazard. Sensitive to static discharge.
Fire Extinguishing Media:
Dry chemical, alcohol foam or carbon dioxide. Water may be ineffective. Water spray may be used to keep fire exposed containers cool, dilute spills to nonflammable mixtures, protect personnel attempting to stop leak and disperse vapors.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and

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unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB® solvent adsorbent is recommended for spills of this product.

---

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
Acetone:
-OSHA Permissible Exposure Limit (PEL):
1000 ppm (TWA)

-ACGIH Threshold Limit Value (TLV):
500 ppm (TWA), 750 ppm (STEL) A4 - not classifiable as a human carcinogen
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded and engineering controls are not feasible, a half-face organic vapor respirator may be worn for up to ten times the exposure limit, or the maximum use concentration specified by the appropriate regulatory agency or respirator

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supplier, whichever is lowest. A full-face piece organic vapor respirator may be worn up to 50 times the exposure limit, or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. For emergencies or instances where the exposure levels are not known, use a full-face piece positive-pressure, air-supplied respirator. WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.

Skin Protection:

Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Eye Protection:

Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

Appearance:

Clear, colorless, volatile liquid.

Odor:

Fragrant, mint-like

Solubility:

Miscible in all proportions in water.

Specific Gravity:

0.79 @ 20C/4C

pH:

No information found.

% Volatiles by volume @ 21C (70F):

100

Boiling Point:

56.5C (133F) @ 760 mm Hg

Melting Point:

-95C (-139F)

Vapor Density (Air=1):

2.0

Vapor Pressure (mm Hg):

400 @ 39.5C (104F)

Evaporation Rate (BuAc=1):

ca. 7.7

---

10. Stability and Reactivity

Stability:

Stable under ordinary conditions of use and storage.

Hazardous Decomposition Products:

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Carbon dioxide and carbon monoxide may form when heated to decomposition.

Hazardous Polymerization:

Will not occur.

Incompatibilities:

Concentrated nitric and sulfuric acid mixtures, oxidizing materials, chloroform, alkalis, chlorine compounds, acids, potassium t-butoxide.

Conditions to Avoid:

Heat, flames, ignition sources and incompatibles.

---

11. Toxicological Information

Oral rat LD50: 5800 mg/kg; Inhalation rat LC50: 50,100mg/m3; Irritation eye rabbit, Standard Draize, 20 mg severe; investigated as a tumorigen, mutagen, reproductive effector.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Acetone (67-64-1)	No	No	None

---

12. Ecological Information

Environmental Fate:

When released into the soil, this material is expected to readily biodegrade. When released into the soil, this material is expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material is expected to readily biodegrade. When released to water, this material is expected to quickly evaporate. This material has a log octanol-water partition coefficient of less than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material may be moderately degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material may be moderately degraded by photolysis. When released into the air, this material is expected to be readily removed from the atmosphere by wet deposition.

Environmental Toxicity:

This material is not expected to be toxic to aquatic life. The LC50/96-hour values for fish are over 100 mg/l.

---

13. Disposal Considerations

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Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

---

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: ACETONE
Hazard Class: 3
UN/NA: UN1090
Packing Group: II
Information reported for product/size: 188L

International (Water, I.M.O.)

Proper Shipping Name: ACETONE
Hazard Class: 3
UN/NA: UN1090
Packing Group: II
Information reported for product/size: 188L

---

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Acetone (67-64-1)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Acetone (67-64-1)	Yes	Yes	No	Yes

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```
  --------\Federal, State & International Regulations - Part 1\--------
--------
                                          -SARA 302-    ------SARA
313------
  Ingredient                              RQ    TPQ     List
Chemical Catg.
  ----------------------------------------  ---   -----   ----  ------
--------
  Acetone (67-64-1)                       No    No      Yes
No

  --------\Federal, State & International Regulations - Part 2\--------
--------
                                                        -RCRA-    -
TSCA-
  Ingredient                              CERCLA    261.33     8(d)
  ----------------------------------------  ------    ------    -----
-
  Acetone (67-64-1)                       5000       U002       No


Chemical Weapons Convention:  No     TSCA 12(b):  No     CDTA:  Yes
SARA 311/312:  Acute: Yes      Chronic: No   Fire: Yes Pressure: No
Reactivity: No         (Pure / Liquid)
```

Australian Hazchem Code: 2[Y]E
Poison Schedule: None allocated.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL IF SWALLOWED OR INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM.
Label Precautions:
Keep away from heat, sparks and flame.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor.
Avoid contact with eyes, skin and clothing.
Label First Aid:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT

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INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. In all cases, get medical attention.

Product Use:
Laboratory Reagent.

Revision Information:
No Changes.

Disclaimer:
****
Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.
****

Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

UNCONTROLLED DOCUMENT

MSDS Number: **M2015** * * * * * *Effective Date:* ***08/10/04*** * * * * * *Supercedes:* ***11/12/01***

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

METHYL ALCOHOL

1. Product Identification

Synonyms: Wood alcohol; methanol; carbinol
CAS No.: 67-56-1
Molecular Weight: 32.04
Chemical Formula: CH3OH
Product Codes:
J.T. Baker: 5217, 5370, 5595, 5794, 5811, 5842, 5869, 9049, 9063, 9065, 9066, 9067, 9069, 9070, 9071, 9073, 9075, 9076, 9077, 9091, 9093, 9096, 9097, 9098, 9263, 9822, 9830, V654
Mallinckrodt: 3004, 3006, 3016, 3017, 3018, 3024, 3041, 3701, 4295, 5160, 8814, H080, H488, H603, H985, V079, V571

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent Hazardous

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Methyl Alcohol	67-56-1	100%	Yes

3. Hazards Identification

Emergency Overview

POISON! DANGER! VAPOR HARMFUL. MAY BE FATAL OR CAUSE BLINDNESS IF SWALLOWED. HARMFUL IF INHALED OR ABSORBED THROUGH SKIN. CANNOT BE MADE NONPOISONOUS. FLAMMABLE LIQUID AND VAPOR. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM AND LIVER.

SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Poison)
Flammability Rating: 3 - Severe (Flammable)
Reactivity Rating: 1 - Slight
Contact Rating: 3 - Severe (Life)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

Inhalation:
A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Once absorbed into the body, it is very slowly eliminated. Symptoms of overexposure may include headache, drowsiness, nausea, vomiting, blurred vision, blindness, coma, and death. A person may get better but then worse again up to 30 hours later.

Ingestion:
Toxic. Symptoms parallel inhalation. Can intoxicate and cause blindness. Usual fatal dose: 100-125 milliliters.

Skin Contact:
Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure.

Eye Contact:
Irritant. Continued exposure may cause eye lesions.

Chronic Exposure:
Marked impairment of vision has been reported. Repeated or prolonged exposure may

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cause skin irritation.

Aggravation of Pre-existing Conditions:

Persons with pre-existing skin disorders or eye problems or impaired liver or kidney function may be more susceptible to the effects of the substance.

---

4. First Aid Measures

Inhalation:

Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

Ingestion:

Induce vomiting immediately as directed by medical personnel. Never give anything by mouth to an unconscious person. Get medical attention immediately.

Skin Contact:

Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.

Eye Contact:

Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

---

5. Fire Fighting Measures

Fire:

Flash point: 12C (54F) CC
Autoignition temperature: 464C (867F)
Flammable limits in air % by volume:
lel: 6.0; uel: 36
Flammable Liquid and Vapor!

Explosion:

Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Moderate explosion hazard and dangerous fire hazard when exposed to heat, sparks or flames. Sensitive to static discharge.

Fire Extinguishing Media:

Use alcohol foam, dry chemical or carbon dioxide. (Water may be ineffective.)

Special Information:

In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Use water spray to blanket fire, cool fire exposed containers, and to flush non-ignited spills or vapors away from fire. Vapors can flow along surfaces to distant ignition source and flash back.

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6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB® solvent adsorbent is recommended for spills of this product.

---

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product. Do Not attempt to clean empty containers since residue is difficult to remove. Do not pressurize, cut, weld, braze, solder, drill, grind or expose such containers to heat, sparks, flame, static electricity or other sources of ignition: they may explode and cause injury or death.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
For Methyl Alcohol:
- OSHA Permissible Exposure Limit (PEL):
200 ppm (TWA)
- ACGIH Threshold Limit Value (TLV):
200 ppm (TWA), 250 ppm (STEL) skin
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document,

Industrial Ventilation, A Manual of Recommended Practices, most recent edition, for details. Use explosion-proof equipment.

Personal Respirators (NIOSH Approved):

If the exposure limit is exceeded and engineering controls are not feasible, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. Breathing air quality must meet the requirements of the OSHA respiratory protection standard (29CFR1910.134). This substance has poor warning properties.

Skin Protection:

Rubber or neoprene gloves and additional protection including impervious boots, apron, or coveralls, as needed in areas of unusual exposure.

Eye Protection:

Use chemical safety goggles. Maintain eye wash fountain and quick-drench facilities in work area.

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9. Physical and Chemical Properties

Appearance:
Clear, colorless liquid.

Odor:
Characteristic odor.

Solubility:
Miscible in water.

Specific Gravity:
0.8

pH:
No information found.

% Volatiles by volume @ 21C (70F):
100

Boiling Point:
64.5C (147F)

Melting Point:
-98C (-144F)

Vapor Density (Air=1):
1.1

Vapor Pressure (mm Hg):
97 @ 20C (68F)

Evaporation Rate (BuAc=1):
5.9

---

10. Stability and Reactivity

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Stability:
Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products:
May form carbon dioxide, carbon monoxide, and formaldehyde when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Strong oxidizing agents such as nitrates, perchlorates or sulfuric acid. Will attack some forms of plastics, rubber, and coatings. May react with metallic aluminum and generate hydrogen gas.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

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11. Toxicological Information

Methyl Alcohol (Methanol) Oral rat LD50: 5628 mg/kg; inhalation rat LC50: 64000 ppm/4H; skin rabbit LD50: 15800 mg/kg; Irritation data-standard Draize test: skin, rabbit: 20mg/24 hr. Moderate; eye, rabbit: 100 mg/24 hr. Moderate. Investigated as a mutagen, reproductive effector.

--------\Cancer Lists\----------

Ingredient	NTP Carcinogen - Known	NTP Carcinogen - Anticipated	IARC Category
Methyl Alcohol (67-56-1)	No	No	None

---

12. Ecological Information

Environmental Fate:
When released into the soil, this material is expected to readily biodegrade. When released into the soil, this material is expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into the water, this material is expected to have a half-life between 1 and 10 days. When released into water, this material is expected to readily biodegrade. When released into the air, this material is expected to exist in the aerosol phase with a short half-life. When released into the air, this material is expected to be readily degraded by reaction with photochemically produced hydroxyl radicals. When released into air, this material is expected to have a half-life between 10 and 30 days. When released into the air, this material is expected to be readily removed from the atmosphere by wet deposition.

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Environmental Toxicity:
This material is expected to be slightly toxic to aquatic life.

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13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

---

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: METHANOL
Hazard Class: 3
UN/NA: UN1230
Packing Group: II
Information reported for product/size: 358LB

International (Water, I.M.O.)

Proper Shipping Name: METHANOL
Hazard Class: 3, 6.1
UN/NA: UN1230
Packing Group: II
Information reported for product/size: 358LB

---

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Methyl Alcohol (67-56-1)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

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```
                                                                  --Canada--
  Ingredient                                              Korea  DSL   NDSL
Phil.
  ----------------------------------------------  -----  ---   ----  -
----
  Methyl Alcohol (67-56-1)                                Yes   Yes   No
Yes

  --------\Federal, State & International Regulations - Part 1\--------
--------
                                                  -SARA 302-    ------SARA
313------
  Ingredient                                      RQ    TPQ     List
Chemical Catg.
  ----------------------------------------  ---   -----   ----  ------
--------
  Methyl Alcohol (67-56-1)                        No    No      Yes
No

  --------\Federal, State & International Regulations - Part 2\--------
--------
                                                                -RCRA-    -
TSCA-
  Ingredient                                      CERCLA     261.33     8(d)
  ----------------------------------------  ------     ------    -----
-
  Methyl Alcohol (67-56-1)                        5000       U154       No


Chemical Weapons Convention:  No     TSCA 12(b):  No     CDTA:  No
SARA 311/312:  Acute: Yes      Chronic: Yes  Fire: Yes Pressure: No
Reactivity: No          (Pure / Liquid)
```

Australian Hazchem Code: 2PE
Poison Schedule: S6
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

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16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
POISON! DANGER! VAPOR HARMFUL. MAY BE FATAL OR CAUSE BLINDNESS IF SWALLOWED. HARMFUL IF INHALED OR ABSORBED THROUGH SKIN. CANNOT BE MADE NONPOISONOUS. FLAMMABLE LIQUID AND VAPOR. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM AND LIVER.
Label Precautions:
Avoid breathing vapor.

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Avoid contact with eyes, skin and clothing.
Wash thoroughly after handling.
Keep container closed.
Use only with adequate ventilation.
Keep away from heat, sparks and flame.

Label First Aid:

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. If swallowed, induce vomiting immediately as directed by medical personnel. Never give anything by mouth to an unconscious person. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. In all cases get medical attention immediately.

Product Use:

Laboratory Reagent.

Revision Information:

No Changes.

Disclaimer:

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Phone Number: (314) 654-1600 (U.S.A.)

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EFFECTIVE DATE: 02/2007 SOP# 401 REVISION # 2

PRESSURIZED FLUID EXTRACTION (ACCELERATED SOLVENT EXTRACTION)

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1.0 Applicable Analytical Methods

1.1 Pressurized Fluid Extraction (PFE), U.S. Environmental Protection Agency, Method 3545, SW846, Revision 0, December 1996.

1.2 Organic Extraction and Sample Preparation, U.S. Environmental Protection Agency, Method 3500B, SW846, Revision 2, December 1996

2.0 Matrix or Matrices

2.1 Soil.

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2.2 Sediment.

2.3 Clay.

2.4 Sludge.

2.5 Waste solids.

3.0 Method Detection Limits

3.1 All method detection limits (MDL) shall be performed according to the Code of Federal Regulations 40 CFR, Part 136, Appendix B. See internal procedure SOP 103 and Section 12.8 of SOP 401.

3.2 Sand is the matrix used for MDL determinations.

3.3 Eight spiked replicates are prepared.

3.4 MDLs are performed annually.

3.5 Blanks are prepared with surrogates in sand.

4.0 Scope and Application

4.1 Method 3545 is used in the extraction of semivolatile organic compounds, organochlorine pesticides, and PCBs from soils, clays, sediments, sludges, and waste solids.

4.2 Elevated temperature (100°C) and pressure (1500 – 2000 psi) are utilized to achieve analyte recovery.

4.3 See DEQ Laboratory SOP 500 for the base neutral acid compound list.

4.4 See DEQ Laboratory SOP 502 for the pesticide, chlorinated hydrocarbon, and PCB compound lists.

4.5 See DEQ Laboratory SOP 507 for the diesel range organics (DRO).

4.6 This method is applicable to solid samples only and is most effective on dry samples with small particle sizes.

4.7 Samples containing large amounts of oil should be extracted using another technique as they clog tubing in the extractor.

5.0 Method Summary

5.1 Standing water is poured off all samples (with the exception of sludge samples). Samples are then stirred to homogenize.

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5.2 Samples are weighed, mixed with diatomaceous earth, transferred to an extraction cell, and spiked with surrogates.

5.3 The accelerated solvent extractor by Dionex (ASE200) program is run and extracted with solvents.

5.4 Extracts are dried with sodium sulfate and concentrated.

5.5 Extracts may be cleaned up using gel permeation chromatography (GPC). See internal SOP 400.

5.6 A silica gel chromatography cleanup can be performed for pesticide and PCB analysis if needed. See internal SOP 402.

6.0 Definitions

6.1 Surrogate analyte (SA) - A pure analyte which is extremely unlikely to be found in any sample and which is added to a sample aliquot in known amounts before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method performance with each sample.

6.2 Method preparation blank (MPB) - An aliquot of a blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and surrogates that are used with samples.

6.3 Laboratory control sample (LCS) - An aliquot of blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is treated exactly as a sample including extraction and analysis.

6.4 Matrix spike (MS) - An aliquot of a sample to which known quantities of the method analytes are added in the laboratory. The MS is treated exactly as a sample including extraction and analysis. The background concentrations of the analytes in the sample must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

6.5 Matrix spike duplicate (MSD) - A second aliquot of sample to which known quantities of the target analytes are added in the laboratory. The MSD is treated exactly as a sample including extraction and analysis. The background concentrations of the analytes in the sample must be determined in a separate aliquot and the measured values in the MSD corrected for background concentrations. Analyses of the MS and MSD indicate precision associated with laboratory procedures.

7.0 Interferences

7.1 All glassware and hardware must be thoroughly cleaned to remove sample artifacts and/or interferences.

7.2 All solvent lots should be tested to ensure the absence of contaminants.

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7.3 Interferences co-extracted from samples can often be removed by cleanup techniques. See internal SOPs 400 and 402.

7.4 The use of plastic must be avoided for semivolatile analyses to prevent phthalate ester contamination.

7.5 Soap residue must be rinsed from all glassware and hardware to avoid degradation of several pesticide compounds.

8.0 Safety

8.1 Safety glasses are required in all designated laboratory areas.

8.2 Be familiar with the Laboratory Chemical Hygiene Plan.

8.3 Be familiar with the Laboratory Safety Policy (SOP 100).

8.4 Gas tanks must be secured to a wall or counter top.

8.5 Extraction cells in the ASE200 oven are hot. Allow cells to cool before handling.

8.6 During the gas purge step, some solvent vapors may exit through the vent port in the instrument. This vent port needs to have the vapor recaptured.

8.7 Protective gloves must be worn when handling samples and solvents.

8.8 Methylene chloride, methanol, phosphoric acid, diatomaceous earth (powder and pelletized), sodium sulfate, hexane, and acetone material safety data sheets (MSDSs) are attached.

9.0 Equipment and Supplies

9.1 Dionex ASE200.

9.2 Extraction cells – 22 mL and 33 mL with collars and frits made of stainless steel and capable of withstanding pressures of 2000 plus psi. See attachment 22.9.

9.3 Dionex solvent controller.

9.4 Computer - Auto ASE software.

9.5 FEP teflon filling funnel - 450mL (Big Science, Inc., part #P00007 or equivalent).

9.6 Turbovap tubes – Caliper, 250 mL.

9.7 Turbovap concentration workstation – Temperature control up to 95°C, pressure from 0 to 20 psi, and sensors set at 0.5 mL.

9.8 Culture tubes – 16 x 100 mm, straight top, VWR part #60825-618.

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9.8.1 For use on GPCs - Fill each tube with 1, 2 or 10 mL of hexane from a calibrated bottletop dispenser. (See 9.22)

9.8.2 Mark the meniscus on each tube insert.
9.8.3 Discard the hexane. Turn the tube upside down in a rack to dry.
9.8.4 For some analysis – Fill previously marked tube (Section 9.8.1) with 10 mL hexane.
9.8.5 Mark a second meniscus on each tube.
9.8.6 Discard the hexane. Turn the tube upside down in a rack to dry.

NOTE: *You will need tubes marked at 1 and 10 mL, 2 mL only, 2 mL and 10 mL, and 10 mL only.*

9.9 Filter – cellulose, 19.1 mm, Type D28 (Dionex), or equivalent, part #049458.

9.10 Foil.

9.11 Beakers – glass: 150 mL or 250 mL.

9.12 Spatulas – metal or ceramic.

9.13 Vials – 2 mL crimp cap, HP #5181-3375.

9.13.1 Place 1 mL of hexane into each vial from a calibrated bottle top dispenser. (Section 9.9). Check the dispenser with a volumetric flask prior to use.
9.13.2 Mark the meniscus with a lab marker.
9.13.3 Dispose of the hexane. Let vial air dry.
9.13.4 Hexane may also be saved for reuse. Transfer used hexane to the container labeled "for pre-marking only."

9.14 Crimp caps – HP #5181-1210.

9.15 Pasteur pipettes – disposable.

9.16 Glass wool. Corning 3950, 8 micron fiberglass.

9.17 Vials - glass screw cap, 60 mL with teflon septum.

9.18 Oven – capable of maintaining at least 130°C.

9.19 Analytical balance – capable of weighing to 0.01 g.

9.20 Syringes – various sizes.

9.21 Bottles - amber screw cap, 250 mL with teflon lined caps.

9.22 Bottletop solvent dispensers – adjustable.

9.22.1 Calibrate to desired volume by filling appropriate volumetric flasks and adjusting dispenser.

9.23 Volumetric flasks – class A, various sizes.

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9.24 Cap – plug tite caps, large; Elkay Products, Inc.; part #127-0019-200.

9.25 Soxtec – System HT2, 1045 extraction unit.

9.26 Snap Cap - Caliper "star" cut snap cap, part #ZB7216.

9.27 Screw cap vials – 13 x 100 mm, Corning, part #99449-13 or equivalent.

9.27.1 Fill each tube with 2 or 5 mL of hexane from a calibrated bottletop dispenser - adjustable. (See 9.21)
9.27.2 Mark the meniscus on each tube.
9.27.3 Discard the hexane. Turn the tube upside down in a rack to dry.

Note: Occasionally 16 x 100 vials are premarked at 10 mL by the same method.

9.28 Screw caps – Corning, part #99999-13 (VWR part #60827-110) or equivalent.

9.29 ASE cell funnels - Dionex #46958.

9.30 Muffle furnace – capable of maintaining 450°C.

9.31 Branson model #1210 ultrasonic cleaner or equivalent.

9.32 Pipets – Class A, various sizes.

9.33 Lancer Laboratory Glassware Washer – Model 1600 UP or equivalent.

9.34 Refrigerator capable of maintaining <6°C. See SOP 127.

9.35 Freezer capable of maintaining $\leq$-10°C. See SOP 127.

10.0 Reagents and Standards

10.1 All reagents, solvents, and standards must be traceable to the stock inventory tracking log. (See Attachment 22.4)

10.2 All reagents, solvents, and standards must be labeled with the date received, the date opened, the expiration date, the tracking number, and the receiver's initials.

10.3 All prepared reagents and standards must be labeled with the date prepared, the expiration date, the preparer's initials, the tracking number, the diluent, and the description. (See Attachment 22.5)

10.4 All standard logbooks must be completely filled out.

10.5 All certificates of analysis must include the stock inventory tracking number that was assigned to the standard.

10.6 Sodium sulfate (ACS) – granular, anhydrous; store in glass containers at 130°C for a minimum of 12 hours prior to use.

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10.7 Solvents – pesticide residue analysis grade, n-hexane, acetone, methylene chloride, and methanol.

10.8 Technical grade acetone (for glassware washing)

10.9 Nitrogen – technical grade or better for Turbovap concentration workstation.

10.10 Nitrogen – Grade 5, 99.999% ultra high purity for ASE.

10.11 Diatomaceous earth (powder for semi-volatiles and PCB/DROs) – JT Baker #1939-01 or equivalent.

10.12 Hydromatrix (pelletized diatomaceous earth for pesticides/PCBs) – Varian part #198004.

10.13 Sand – free of interferences. Muffled at 450°C overnight. Stored at 105°C or higher until used.

10.14 Surrogate solutions

10.14.1 Base neutral (BN) surrogate – (for semi-volatile analysis)Absolute Standards #24016 or equivalent, 5000 ug/mL in methylene chloride.
2-Fluorobiphenyl
Nitrobenzene-d5
p-Terphenyl-d14

10.14.2 Acid surrogate - Absolute Standards #24015 or equivalent, 10,000 ug/mL in methanol.
2-Fluorophenol
Phenol-d6
2,4,6-Tribromophenol

10.14.3 Semi-volatile surrogate solution (BN = 50 ppm, A = 100 ppm)

10.14.3.1 Partially fill a 500 mL volumetric flask with methanol.
10.14.3.2 Pipette 5 mL of BN surrogate (Section 10.14.1) into the flask.
10.14.3.3 Pipette 5 mL of acid surrogate (Section 10.14.2) into the flask.
10.14.3.4 Dilute the flask to the mark with methanol. Mix.
10.14.3.5 Transfer to amber screw cap bottles. Label.
10.14.3.6 Store solution in a freezer. Replace every six months or sooner as needed.

10.14.4 Pesticide/PCB surrogate solution (1 mg/L)

10.14.4.1 Absolute Standards #20023 or equivalent, 200 ppm in acetone.
Decachlorobiphenyl
2,4,5,6-Tetrachloro-m-xylene.
10.14.4.2 Partially fill a 200 mL volumetric flask with methanol.
10.14.4.3 Inject 1000 uL of stock surrogate solution (Section 10.14.4.1) directly into the methanol in the flask.
10.14.4.4 Dilute the flask to the mark with methanol. Mix.
10.14.4.5 Transfer to amber screw cap bottles. Label.

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10.14.4.6 Store solution in a freezer. Replace every six months or sooner as needed.

10.14.5 PCB surrogate (when extracted with DRO) (2mg/L).

10.14.5.1 Absolute Standards #20023 or equivalent, 200 ppm in acetone.
Decachlorobiphenyl
2,4,5,6-Tetrachloro-m-xylene.
10.14.5.2 Partially fill a 100 mL volumetric flask with methanol.
10.14.5.3 Inject 1000 uL of stock surrogate solution (Section 10.14.4.1) directly into the methanol in the flask.
10.14.5.4 Dilute the flask to the mark with methanol. Mix.
10.14.5.5 Transfer to amber screw cap bottles. Label.
10.14.5.6 Store solution in a freezer. Replace every six months or sooner as needed.

10.14.6 DRO Surrogate Solution.

10.14.6.1 n-Eicosane – purchased as neat (Ultra Scientific RNA-011 or similar)
10.14.6.2 Intermediate standard (10,000 µg/mL). In a 10 mL volumetric flask, add 0.1g neat material. Dilute to volume with acetone.
10.14.6.3 Working standard (20 mg/L). In a 500 mL volumetric flask, add 1 mL of 10,000 µg/mL standard. Dilute to volume with acetone. Mix.
10.14.6.4 Transfer to amber screw cap bottles. Label.
10.14.6.5 Store solution in a freezer. Replace every six months or sooner as needed.

10.15 Spike Solutions

10.15.1 Semi-volatile spike solution (BN = 50 ug/mL, A = 100 ug/mL)

10.15.1.1 Semi-volatiles are spiked with the full list using a spike prepared commercially. Absolute part #94206 or equivalent in methanol. See attachment 22.19.

10.15.2 Pesticide spike solution

10.15.2.1 Accustandard #S-7179 or equivalent, 1000 mg/L in methanol.

Aldrin	Endosulfan I
a-BHC	Endosulfan II
b-BHC	Endosulfan sulfate
d-BHC	Endrin
g-BHC (lindane)	Endrin aldehyde
BP-6 (PBB)	Endrine ketone
a-Chlordane	Heptachlor
g-Chlordane	Heptachlor epoxide
4,4'-DDD	Hexabromobenzene
4,4'-DDE	Methoxychlor
4,4'-DDT	Mirex
Dieldrin	

10.15.2.2 Pesticide spike solution, 1000 ug/L

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10.15.2.2.1 Partially fill a 200 mL volumetric flask with methanol.
10.15.2.2.2 Inject 200 uL of stock pesticide (Section 10.15.2.1) directly into the methanol in the flask.
10.15.2.2.3 Dilute to the mark with methanol. Mix.
10.15.2.2.4 Transfer to 250 mL amber screw cap bottles. Label.
10.15.2.2.5 Store in a freezer. Replace every six months or sooner as needed.
10.15.2.2.6 Transfer the remainder of the contents of the stock pesticide ampule (Section 10.15.2.1) to a crimp cap vial. Label. Store in a freezer.

10.15.3 PCB spike solution (Aroclors 1016 and 1260)

10.15.3.1 PCB mix – Absolute Standards #20064, 1000 ppm in hexane or equivalent Aroclor 1016/1260 mix.
10.15.3.2 PCB spike solution (Aroclor 1016, 5 ppm and Aroclor 1260, 5 ppm)

10.15.3.2.1 Partially fill a 100 mL volumetric flask with methanol.
10.15.3.2.2 Inject 500 µL of Aroclor 1016/1260 mix (Section 10.15.3.1) directly into the methanol in the flask.
10.15.3.2.3 Dilute the flask to the mark with methanol. Mix.
10.15.3.2.4 Transfer to amber screw cap bottles. Label.
10.15.3.2.5 Store solution in a freezer. Replace every six months or sooner as needed.

10.15.3.3 PCB spike solution (when extracted with DRO) (10 ppm each).

10.15.3.3.1 Partially fill a 50 mL volumetric flask with methanol.
10.15.3.3.2 Inject 500 µL of Aroclor 1016/1260 mix (Section 10.15.3.1) directly into the methanol in the flask.
10.15.3.3.3 Dilute the flask to the mark with methanol. Mix.
10.15.3.3.4 Transfer to amber screw cap bottles. Label.
10.15.3.3.5 Store solution in a freezer. Replace every six months or sooner as needed.

10.15.4 DRO spike solution.

10.15.4.1 Diesel fuel/Motor Oil (2000/4000 µg/mL)
10.15.4.2 In a 25 mL volumetric flask, add 500 µL of 100 mg/mL diesel (refer to SOP 507 Section 10.11.2.2) to 1000 µL of 100 mg/mL Oil (refer to SOP 507 Section 10.11.3.2). Dilute to volume with acetone. Mix.
10.15.4.3 Transfer to amber screw cap bottles. Label.
10.15.4.4 Store solution in a freezer. Replace every six months or sooner as needed.

10.16 Phosphoric acid (H_3PO_4) – Baker #0260-3 or equivalent.

10.17 DI water – Organic free.

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10.18 Semi-volatile extraction solvent

10.18.1 Mix 600 mL of acetone (Section 10.7) with 400 mL of methylene chloride (Section 10.7).
10.18.2 Add 20 mL of DI H_2O (Section 10.17).
10.18.3 Add 4 mL of phosphoric acid (Section 10.16). Mix. Place in bottle "C" on the ASE.

10.19 Helium – high purity for sparging GPC mobile phase.

10.20 Lancer Acid for Glasswasher (LCA 30 GL or equivalent) Raush and Associates. 25% glacial acetic acid, 75% water.

10.21 Lancer Clean for Glasswasher (LCD 30 GL or equivalent) Raush and Associates.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 An 8-ounce, wide mouth, glass jar sealed with aluminum-foil lined screw caps is the recommended sample container to be used.

11.2 Samples should be stored in ice until the Laboratory receives them. They are then refrigerated at 4°C until extraction.

11.3 The U.S. Environmental Protection Agency maximum holding time is 14 days from sample collection to extraction.

11.4 Sample extracts should be stored in a freezer and must be analyzed within 40 days of extraction.

12.0 Quality Control (QC)

12.1 Temperature is monitored daily. See attachment 22.16. See SOP 127 for corrective action.

12.1.1 Room temperature for Room 302.
12.1.2 Freezers Room 302 and 302B.
12.1.3 Refrigerator Room 302.
12.1.4 Oven Room 302 and 302A.

12.2 Balances are calibrated at least once per year by an outside source.

12.2.1 Balance calibrations are checked weekly and recorded in the balance check logbook. See attachment 22.18; see SOP 128 for corrective action.
12.2.2 Calibration weights are certified by an outside source. See SOP 128.

12.3 Sample batches consist of a maximum of 20 samples, one method preparation blank, one MS, one MSD, one LCS, and one LCSD.

12.4 MSs and MSDs are actual samples that are selected at random from within the batch of 20. They may also be client designated.

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12.5 All 20 samples and the QC samples are spiked with known surrogate concentrations. The recoveries for these compounds are used as indicators of possible problems that may occur during extraction and cleanup of individual samples.

12.6 The MSs, MSDs, LCSs and LCSDs are spiked at a known concentration with the analyte compounds. The replicate matrix spikes are used to evaluate the entire batch for precision and accuracy.

12.7 Initial demonstration of capability (IDOC) – Each analyst must demonstrate initial proficiency with this method by generating data of acceptable accuracy and precision for target analytes.

12.7.1 Assemble five extraction cells as in Section 14.1.1 using the 22 mL cells for pesticides/PCBs or the 33 mL cells for semi-volatiles and PCB/DROs.

12.7.2 Prepare samples as in 14.1.3.1-7, using 10 g of sand for pesticide/PCBs and 25 g of sand for semi-volatiles and PCB/DROs.

12.7.3 Spike all 5 cells with 1 mL of semi-volatile surrogate (Section 10.14.3), 1 mL of pesticide/PCB surrogate (Section 10.14.4) or 1 mL each of PCB surrogate (10.14.5) and DRO surrogate (10.14.6) depending on the target compounds.

12.7.4 Spike 4 cells with 1 mL of semi-volatile spike (Section 10.15.1), 1 mL of pesticide spike (Section 10.15.2.2), 1 mL of PCB spike (Section 10.15.3.2) or 1 mL each of PCB spike (10.15.3) and DRO spike (10.15.4) depending on the target compounds. Label the un-spiked cell "MPB."

12.7.5 Place enough diatomaceous earth (Section 10.11 or 10.12) in each of the five cells to fill. Screw an end cap onto each cell.

12.7.6 Proceed with Sections 14.1.6 through 14.1.8.

12.7.7 Follow all cleanup procedures used for routine samples.

12.7.8 Analyze and calculate recoveries by the same procedures used to analyze samples (SOPs 500 or 502).

12.8 MDLs

NOTE: Number of cells prepared will vary depending upon number of ASEs and GPCs used.

12.8.1 Prepare MDLs at levels specified by the analyst and with spike solutions they provide.

12.8.2 Assemble twelve extraction cells as in Section 14.1.1, using the 22 mL cells for pesticides/PCBs and the 33 mL cells for semi-volatiles and PCB/DROs.

12.8.3 Prepare samples as in 14.1.3, using 10 g of sand for pesticide/PCBs and 25 g of sand for semi-volatiles and PCB/DROs.

12.8.4 Spike all twelve cells with 1 mL of semi-volatile surrogate (Section 10.14.3), 1 mL of pesticide/PCB surrogate (Section 10.14.4) or 1 mL each of PCB surrogate (10.14.5) and DRO surrogate (10.14.6) depending on the target compounds.

12.8.5 Spike eight cells with 1 mL of spike provided by the analyst. Label the un-spiked cells "MPB."

12.8.6 Place enough diatomaceous earth (Section 10.11or 10.12) in each of the twelve cells to fill. Screw an end cap onto each cell.

12.8.7 Proceed with Sections 14.1.6 through 14.1.8, using both ASE instruments and splitting samples evenly between GPCs.

12.8.8 Follow all cleanup procedures used for routine samples, splitting samples evenly between GPCs.

12.8.9 Analyze and calculate recoveries by the same procedures used to analyze samples (SOPs 500 or 502).

13.0 Calibration and Standardization

13.1 This section is not applicable to this method.

14.0 Procedure

14.1 ASE200

14.1.1 Assembly of ASE200 extraction cells

14.1.1.1 Stainless steel frits are placed into the recessed area of the end cap and secured into place by the PEEK seal and cap insert. The entire assembly is locked into place by a snap ring (See Attachment 22.9).

14.1.1.2 One end cap is screwed onto the bottom of the cell body (see Attachment 22.9). Push a filter (Section 9.9) through the cell body to rest on the frit (see Attachment 22.10). Use the 22 mL cells for pesticide/PCB analysis and the 33 mL cells for semi-volatile analysis. A small amount of diatomaceous earth (section 10.11 or 10.12) is added to cell before the sample.

14.1.2 Two hot rinses are used to clean the ASE200 lines at the end of a run. The MPB extraction cell is run as a sample utilizing the hot rinse program (Section 16.1.3). This is the only way to run hot solvent through the lines where possible contamination of polymers may have occurred. Often a blank regular run is also scheduled utilizing the MPB cell for additional cleaning.

14.1.3 Sample preparation

14.1.3.1 Allow the sample to warm to room temperature.

14.1.3.2 Decant and discard any water layer on top of the sample, unless designated a sludge.

14.1.3.3 Stir the sample with a spatula. Weigh 10.0 g for pesticide/PCB extraction or 25.0 g for semi-volatile and PCB/DRO extraction into a 150 or 250 mL beaker.

NOTE: *The sample may be weighed at this time for total solids determination (Section 14.1.5).*

14.1.3.4 Record the sample weight in the ASE runlog to 0.01g (See Attachment 22.3). Label the beaker.

14.1.3.5 Mix the sample with approximately 1 g of diatomaceous earth (Section 10.11 or 10.12). Allow time for the sample to dry. The sample should be stirred occasionally or it may harden.

14.1.3.6 Transfer the sample to an extraction cell. Use the large (33 mL) size for the semi-volatiles and PCB/DROs or the small (22 mL) size for pesticides/PCBs.

14.1.3.7 If the consistency of the matrix is such that it will not fit into the prescribed cell, the weight may be halved or the pesticide/PCB samples may use the larger cells. Document.

14.1.3.8 Add surrogate compounds. Add 1 mL of semi-volatile surrogate (Section 10.14.3), 1 mL of pesticide/PCB surrogate (Section 10.14.4) or 1 mL each of the PCB surrogate (10.14.5) and DRO surrogate (10.14.6) depending upon the extraction type.

14.1.3.9 Fill the empty space that remains with diatomaceous earth (Section 10.11 or 10.12). Tap the cell to settle, but do not pack it tightly. Screw an end cap on the extraction cell.

14.1.4 Quality assurance/quality control (QA/QC) sample preparation.

14.1.4.1 MPB

14.1.4.1.1 Prepare one MPB by weighing 10 or 25 g of sand (depending on the extraction type) into a 150 or 250 mL beaker.

14.1.4.1.2 Record the sample weight in the ASE run log to 0.01 g. Label the beaker.

14.1.4.1.3 Mix the sample with approximately 1 g of diatomaceous earth (Section 10.11 or 10.12).

14.1.4.1.4 Transfer to an extraction cell (33 mL or 22 mL depending on the extraction type).

14.1.4.1.5 Add 1 mL of semi-volatile surrogate (Section 10.14.3), 1 mL of pesticide/PCB surrogate (Section 10.14.4) or 1mL each of the PCB surrogate (10.14.5) and DRO surrogate (10.14.6) depending on the target compounds.

14.1.4.1.6 Fill the empty space that remains with diatomaceous earth (Section 10.11 or 10.12).

14.1.4.1.7 Screw end cap on extraction cell. Label.

14.1.4.2 LCS/LCSD - Prepare two LCSs by following 14.1.4.1.1 through 14.1.4.1.5.

14.1.4.2.1 Add 1 mL of semi-volatile spike (Section 10.15.1), 1 mL of pesticide spike (Section 10.15.2.2), 1 mL of PCB spike (Section 10.15.3.2) or 1 mL each of PCB spike (10.15.3.3) and DRO spike (10.15.4) depending on the target compounds.

14.1.4.2.2 Fill the empty space that remains with diatomaceous earth (Section 10.11 or 10.12). Screw an end cap on the extraction cell.

14.1.4.2.3 Screw end cap on extraction cell. Label.

14.1.4.3 MS/MSD/matrix sample

14.1.4.3.1 Prepare three replicates of a sample in the batch by weighing 40 g (pesticide/PCB) or 80 g (semi-volatile or PCB/DRO) sample into a 250 mL beaker depending upon the target analytes. This is to obtain homogenous samples.

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14.1.4.3.2 Stir the sample with a spatula. Weigh three aliquots of 10.0 g for pesticide/PCB extraction, or 25.0 g for semi-volatile or PCB/DRO extraction into three separate 150 or 250 mL beakers.

14.1.4.3.3 Follow Sections 14.1.3.4 through 14.1.3.8.

14.1.4.3.4 Add 1 mL of semi-volatile spike (Section10.15.1), 1 mL of pesticide spike (Section 10.15.2.2), 1 mL of PCB spike (Section 10.15.3.2) or DRO spike (10.15.4) depending on the target compounds to the two extraction cells (for MS/MSD only).

14.1.4.3.5 The third sample receives the surrogate only. (QC matrix sample)

14.1.4.3.6 Fill the empty space that remains with diatomaceous earth (Section 10.11 or 10.12).

14.1.4.3.7 Screw end caps on all extraction cells.

14.1.5 Percent total solids (% TS) determination (usually done by the Inorganic Unit).

14.1.5.1 Weigh an aluminum pan. Record this weight in the % total solids (% TS) logbook (see Attachment 22.7). Tare the pan.

14.1.5.2 Weigh a 10 g portion of sample into the tarred aluminum pan. Record the sample weight in the % TS logbook.

14.1.5.3 Dry in a 105°C oven overnight. Remove. Place in a desiccator. Cool to room temperature.

14.1.5.4 Weigh the sample and aluminum pan. Record this weight in the logbook.

14.1.5.5 Refer to Section 15.1 for calculations.

14.1.6 Program ASE200

14.1.6.1 With the use of autoase software and a solvent controller, the soft keypad on the ASE200 will reflect that it is on remote. The programs in Section 16.1 are the methods used.

14.1.6.2 Open the nitrogen tank valve. The pressure should be 150 pounds per square inch.

14.1.6.3 Press the “on” button on the ASE200. Wait until item 1 on the menu screen reads “Load Method/Schedule.”

14.1.6.4 Turn the computer on.

14.1.6.5 Double click on the “autoase” icon.

14.1.6.6 The ASE200 screen should indicate that it is idle.

14.1.6.7 There will be two blue bars on the computer screen, one for instrument #17 (system 1) and one for instrument #61 (system 2), both should read “idle.”

14.1.6.8 Load the extraction cells onto the top rack of the ASE to be used.

14.1.6.9 Place 60 mL glass screw cap collection vials (Section 9.16) on the bottom rack.

14.1.6.10 Remove the extraction cell labels and attach them to the corresponding vial in the bottom carousel.

***NOTE**: Do not leave any labels on the extraction cells. Place the labels on the vials facing in toward the carousel or a sensor will give an error reading.*

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14.1.6.11 Fill all solvent bottles needed for the extraction.

14.1.6.12 Empty rinse vials (four) on the bottom of the carousel and one vial inside the ASE200. Empty at least two vials for hot rinses and cleaning rinses.

14.1.6.13 Schedule set up on computer.

14.1.6.13.1 Click on the blue bar for the ASE system you want to program.

14.1.6.13.2 Click “Edit”.

14.1.6.13.3 Click “Schedule File”.

14.1.6.13.4 Click “Open”.

14.1.6.13.5 Click on an existing batch of the same type to be extracted.

14.1.6.13.6 Click “OK”. Click “Save As”. Changes can now be made.

14.1.6.13.7 Type in the new batch number and test code at the “Schedule Name” prompt. Click “OK”.

NOTE: *Vial numbers are not changeable and refer to the collection vials on the bottom carousel. Cell numbers are assignable and refer to the extraction cells on the top carousel. Extraction cells can be assigned to any vial and to more than one vial. Only cells that have been assigned a method will run. Extra cell numbers do not need to be deleted.*

14.1.6.13.8 Method name

14.1.6.13.8.1 Method name should be correct.

14.1.6.13.8.2 If need to change, double click on the method cell to display the dialog box.

14.1.6.13.8.3 Select the desired file. Click “OK”.

14.1.6.13.9 Extraction line rinse – Click the “on” or “on D” button to rinse after the extraction.

14.1.6.13.9.1 Pesticide/PCB rinse – “on“ for rinses with “A” - MeCl.

14.1.6.13.9.2 semi-volatile rinse – “on D” for rinses with D (Section 16.1) 25/25/50 acetone/methanol/H_2O.

14.1.6.13.10 Sample ID - Enter the sample identification for the vial number in the schedule. This can be alphabetical or numeric up to 32 characters.

14.1.6.13.11 The last two samples should be cell #1 and the last two vials. The “hot rinse” method (see Section 16) should be utilized. Sometimes a clean-up run is added also cell #1 and final vial.

14.1.6.13.12 Click “Save As”. The batch number should be visible in the file name bar.

14.1.6.13.13 Click “OK”. Click “Print” to print the schedule.

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14.1.6.13.14 Click “Exit”. The screen should return to “idle.”

14.1.6.14 Load schedule

14.1.6.14.1 Click “File.”
14.1.6.14.2 Click “Load Schedule.” Scroll down the list of file names to the desired batch number. Click on that batch number. (It should now appear in the single line file name.)
14.1.6.14.3 The starting vial should read “1” for the starting extraction. Click “OK”. The idle screen should now have the chosen batch number in the schedule line. The sample method should be in the method line.
14.1.6.14.4 Push the start icon (extraction cell with a down arrow).

14.1.6.15 Shutdown (After all samples have been run)

14.1.6.15.1 Exit out of the auto ASE software.
14.1.6.15.2 Turn the computer off.
14.1.6.15.3 Turn the ASE unit off.
14.1.6.15.4 Turn the nitrogen tank off.

14.1.7 Water removal from extracts

14.1.7.1 Pack a plug of glass wool into a funnel (Section 9.5).
14.1.7.2 Add approximately two inches of cooled sodium sulfate (Section 10.6) to each funnel. Rinse them with methylene chloride until solvent runs through. Discard the rinse solvent.
14.1.7.3 Place enough sodium sulfate in the collection vial to absorb all water.
14.1.7.4 Place a 250 mL Turbovap tube under a funnel. Pour the extract onto the funnel to filter and dry the sample. Transfer the label to the Turbovap tube.

14.1.7.4.1 Rinse the collection vial with approximately 20 mL of methylene chloride. Add this to the funnel.
14.1.7.4.2 Repeat this rinse two more times collecting all rinses in the 250 mL Turbovap tube.

14.1.8 Extract concentration

14.1.8.1 Place the 250 mL Turbovap tube on the Turbovap unit at a temperature of 35°C and a pressure of 15 psi.
14.1.8.2 Evaporate the samples down to 0.5 mL. Transfer extracts for semi-volatile analysis into 10 mL pre-marked culture tubes (Section 9.8) with pasteur pipettes. Transfer extracts for pesticides and/or PCB analysis into 5.0 mL premarked vials (Section 9.27) with pasteur pipettes. Transfer extracts for PCB/DRO analysis into a 1 and 10 mL pre marked GPC culture tube (Section 9.8.6).
14.1.8.3 Rinse the Turbovap tube with methylene chloride. Transfer to the culture tube or vial.

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14.1.8.4 Dilute the tube to the 10 mL pre-marked line and the vial to the 5 mL premarked line with methylene chloride.

14.1.8.4.1 For semi-volatile analysis utilizing the Caliper GPCs, place foil on top of the tube and cap (Section 9.26) if going immediately onto the GPC or a cap (Section 9.24) if storing in the refrigerator.

14.1.8.4.2 For semi-volatile analysis utilizing the J2 Scientific GPCs, bring the volume to 10 mL with MeCl and either cap (Section 9.24) or filter as in SOP 400.

14.1.8.4.3 For pesticide and/or PCB analysis, transfer 2 mL of extract into a culture tube (Section 9.8) if going directly onto a GPC. Place foil on top of tube and cap (Section 9.26) if using Caliper GPCs. See Section 14.1.8.4.4 for J2 Scientific GPCs. Cap the remainder (Section 9.28). Store in refrigerator.

14.1.8.4.4 For pesticide and/or PCB analysis utilizing the J2 Scientific GPCs, bring the volume to 10 mL with MeCL and either cap (Section 9.24) or filter as in SOP 400.

14.1.8.4.5 For PCB/DRO analysis. Mix extract well. Transfer 9 mL back into TurboVap tube for DRO analysis and add "D" to label to indicate 1st transfer has been completed. Remaining 1 mL is taken to 10 mL with methylene chloride and run on GPC with 10 mL program for PCB analysis. Follow semi-volatile 10 mL instructions 14.1.8.4.1 or 14.1.8.4.2.

14.1.8.4.5.1 Extract for DRO analysis (9 mL in TurboVap tube) is concentrated back to 0.5 mL. Transfer to 1 mL pre-marked crimp cap vials (Section 9.13), cap, and label. Make a copy of the ASE run log onto yellow paper.

14.1.8.4.5.2 Place the extracts into the corresponding refrigerators or freezers. Place the paperwork in appropriate bin.

14.1.8.5 Samples are now ready for cleanup methods.

NOTE: *If a sample will not concentrate to approximately 1.0 mL, transfer to a premarked 5 or 10 mL vial (Section 9.27)or volumetric flask (section 9.23) and dilute with methylene chloride. Document well.*

14.2 Glassware cleaning.

14.2.1 Hand washing

14.2.1.1 Rinse beakers and spatulas with warm tap water.

14.2.1.2 Disassemble cells and rinse with warm tap water and MeCl.

14.2.1.3 Rinse Turbovap tubes with MeCl.

14.2.1.4 Beakers, spatulas, cell bodies and caps, and dirty Turbovap tubes need to be washed with hot, soapy water (Liqui-Nox soap).

14.2.1.5 Rinse with hot tap water.
14.2.1.6 Place in dishwasher.

14.2.2 Dishwasher

14.2.2.1 Place solvent rinsed Turbovap tubes, hand washed glassware or cell in dishwasher.
14.2.2.2 Run as in attachment 22.14.

14.2.3 Cell frit and snap ring cleaning

14.2.3.1 Place frits and snap rings into separate beakers. Cover with tap water and run in ultrasonic cleaner for at least 10 minutes.
14.2.3.2 Pour off water and rinse with acetone. Pour off acetone and cover with acetone. Sonicate for at least 10 minutes.
14.2.3.3 Pour off acetone and rinse with MeCl. Pour off MeCl cover with fresh MeCl. Run in the ultrasonic cleaner for at least 10 minutes.
14.2.3.4 Rinse with fresh MeCl. Discard MeCl.
14.2.3.5 Collars are ready to dry in oven.
14.2.3.6 Transfer frits to soxtec beaker and cover with MeCl.
14.2.3.7 Turn on soxtec unit and water. Run for at least 30 minutes.
14.2.3.8 Turn off soxtec unit and water. Pour off MeCl.
14.2.3.9 Transfer frits to another beaker. Rinse with MeCl. Drain and place in oven to dry.

14.2.4 Rinse all glassware with MeCl before using.

15.0 Calculations

15.1 % TS

$$\%\ TS = \frac{[\text{weight of dry sample and pan (Section14.1.5.4)} - \text{weight of pan (Section 14.1.5.1)}] \times 100}{\text{weight of wet sample (Section14.1.5.2)}}$$

16.0 Method Performance

16.1 ASE200 conditions

16.1.1 Pesticide_PCB1(or2).met (Also used for PCB/DRO samples)
Method Editor

Preheat	0 minutes	Pressure	2000 psi
Heat	5.0 minutes	Temperature	100°C
Static	5.0 minutes	Sol. A	50%
Flush %	50 volume	Sol. B	50%
Purge	60 seconds	Sol. C	--
Cycles	1	Sol. D	--

where: Sol. A = methylene chloride.
Sol. B = acetone.
Sol. C = semi-volatile extraction solvent (Section 10.18).
Sol. D = 25/25/50 acetone/methanol/H_2O.

16.1.2 Sediment BNA (or BNA2).met

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Method Editor

Preheat	0 minutes	Pressure	2000 psi
Heat	5.0 minutes	Temperature	100°C
Static	5.0 minutes	Sol. A	0
Flush %	50 volume	Sol. B	0
Purge	60 seconds	Sol. C	100
Cycles	1	Sol. D	0

where: Sol. A = methylene chloride.
Sol. B = acetone.
Sol. C = semi-volatile extraction solvent (Section 10.18).
Sol. D = 25/25/50 acetone/methanol/H_2O.

16.1.3 Hot rinse method.

Method Editor

Preheat	0 minutes	Pressure	2000 psi
Heat	7.0 minutes	Temperature	150°C
Static	5.0 minutes	Sol. A	0
Flush %	60 volume	Sol. B	0
Purge	60 seconds	Sol. C	0
Cycles	1	Sol. D	100

where: Sol. A = methylene chloride.
Sol. B = acetone.
Sol. C = semi-volatile extraction solvent (Section 10.18).
Sol. D = 25/25/50 acetone/methanol/H_2O.

16.2 Nitrogen (ultra high purity) at 150 pounds per square inch.

17.0 Pollution Prevention

17.1 This method uses smaller quantities of organic solvents, and the waste that is produced is collected for disposal as chlorinated waste.

17.2 For information concerning pollution prevention which may be applicable to laboratory operations, consult Less is Better: Laboratory Chemical Management for Waste Reduction available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, DC 20036.

18.0 Data Assessment

18.1 Samples must be extracted within 14 days of collection. If samples are extracted past the holding time, it must be noted in the logbook and on the Laboratory Information Management System (LIMS).

18.2 LIMS entry: Creating a batch.

18.2.1 Select the Labworks CITRIX or sharepoint icon on the desktop.
18.2.2 Enter your User ID and password. Select the ENV_PRD database. Click "OK".
18.2.3 Click "QA Batch".
18.2.4 Select "New Batches", then "Batch by Analysis".
18.2.5 Enter analysis in white box. Either E_SBA, E_PPS, E_DROS. Click "OK".

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18.2.6 Click “OK” again. At this point, a list of possible samples for that analysis will appear. The samples included in this batch need to be marked with a red check. Uncheck those samples that are not in the batch.
18.2.7 Click “OK”.
18.2.8 Make sure that the batch size matches or is greater than the number of samples. Click “OK”.
18.2.9 Change the default batch name to match the extractions batch number by changing the last four numbers only. Example: from default number E_PPS-5111 to actual batch number E_PPS-4577.
18.2.10 Click on sample that is being used for QC. Change QA Group and QA Tests as needed.
18.2.11 Click “OK”.
18.2.12 Click “Exit”.

18.3 LIMS entry: Entering results.

18.3.1 Select the Labworks CITRIX or sharepoint icon on the desktop.
18.3.2 Enter your User ID and password. Select the ENV_PRD database. Click “OK”.
18.3.3 Click “Results”.
18.3.4 Results entry template should read “Extract”. Select “QA/QC Batch”.
18.3.5 Select appropriate analysis code. Either E_SBA, E_PPS, or E_DROS. Click “OK”.
18.3.6 Select appropriate batch. Click “OK” twice.
18.3.7 Enter CO under “Results” and check that the dates and initials are correct. Make any changes on the first sample; all following samples will be the same. Fill across. Add any qualifier codes to the “Qualify” line. Find the QC sample. Scroll down and enter surrogate and spike tracking numbers. Click “Save”.
18.3.8 Click “Exit”.

18.4 Back log reports

18.4.1 Log onto Labworks
18.4.2 Place pointer and click on backlog.
18.4.3 Click “Display Reports”.
18.4.4 Type “o_se”.
18.4.5 Click on the printer icon.
18.4.6 Click “Close”. Click “Yes”. Click “Close”.

19.0 Corrective Actions

19.1 If surrogate recoveries fall outside the acceptance limits, has no obvious matrix interference, and the sample is within the 14-day hold time, re-extraction is performed if the sample volume is sufficient.

19.2 All data reported for samples extracted outside the hold time receives a hold time code.

19.3 Corrective Action Forms (attachment 22.17) are completed when problems/errors are discovered and corrected.

20.0 Waste Management

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20.1 The ASE200 has seven waste vials which are emptied into a waste container marked “chlorinated waste”.

20.2 See SOP 111 for more information.

21.0 References

21.1 United States Code of Federal Regulations, Title 40, U.S. Government Printing Office, Washington, D.C., Appendix A & B.

21.2 Test Methods for Evaluating Solid Waste, U.S. Environmental Protection Agency, Document SW-846, Third Edition, 1986, Final Update III, December 1996.

21.3 ASE200 Accelerated Solvent Extractor Operator’s Manual, Dionex, Document No. 031149, Revision 03, March 1997.

21.4 Auto ASE Software User’s Guide, Dionex, Document No. 031259, Revision 01, March 1997.

22.0 Attachments

22.1 Methylene chloride MSDS.

22.2 Acetone MSDS.

22.3 ASE extraction run logbook page.

22.4 Stock standard logbook page.

22.5 Semivolatile standard logbook page.

22.6 Methanol MSDS.

22.7 Total solids logbook page.

22.8 Hexane MSDS.

22.9 ASE extraction cell assembly.

22.10 Cellulose filter positioning diagram.

22.11 Phosphoric acid MSDS.

22.12 Sodium sulfate MSDS.

22.13 Diatomaceous earth MSDS.

22.14 Dishwasher program.

22.15 Hydromatrix Diatomaceous Earth MSDS.

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22.16 Equipment Monitoring Record

22.17 Corrective Action Form

22.18 Weekly Balance Calibration Check

22.19 Semi-volatile spike.

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Signature Page

APPROVED BY _____ DATE 2/26/07
CHIEF, LABORATORY SERVICES SECTION

APPROVED BY _____ DATE _____
COMPLIANCE OFFICER

APPROVED BY _____ DATE 2-22-07
UNIT MANAGER

APPROVED BY _____ DATE 2/21/07
SECOND SHIFT MANAGER

APPROVED BY _____ DATE 2/21/07
ANALYST

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MSDS Number: **M4420** * * * * * *Effective Date:* ***10/06/05*** * * * * * *Supercedes:* ***05/14/03***

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

J.T.Baker®

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300
National Response in Canada
CANUTEC: 613-996-6666
Outside U.S. And Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

METHYLENE CHLORIDE

1. Product Identification

Synonyms: MC; Dichloromethane (DCM); Methylene dichloride; Methylene bichloride; Methane dichloride
CAS No.: 75-09-2
Molecular Weight: 84.93
Chemical Formula: CH2Cl2
Product Codes: 9235, 9264, 9266, 9295, 9315, 9324, 9329, 9330, 9348, 9350, 9965, Q480

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Methylene Chloride	75-09-2	> 99%	Yes

3. Hazards Identification

Emergency Overview

WARNING! HARMFUL IF SWALLOWED, INHALED OR ABSORBED THROUGH SKIN. AFFECTS CENTRAL NERVOUS SYSTEM, LIVER, CARDIOVASCULAR SYSTEM, AND BLOOD. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. SUSPECT CANCER HAZARD. MAY CAUSE CANCER. Risk of cancer depends on level and duration of exposure.

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SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Cancer Causing)
Flammability Rating: 1 - Slight
Reactivity Rating: 2 - Moderate
Contact Rating: 3 - Severe
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: Blue (Health)

Potential Health Effects

Inhalation:
Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes formation of carbon monoxide in blood which affects cardiovascular system and central nervous system. Continued exposure may cause increased light-headedness, staggering, unconsciousness, and even death. Exposure may make the symptoms of angina (chest pains) worse.
Ingestion:
May cause irritation of the gastrointestinal tract with vomiting. If vomiting results in aspiration, chemical pneumonia could follow. Absorption through gastrointestinal tract may produce symptoms of central nervous system depression ranging from light headedness to unconsciousness.
Skin Contact:
Causes irritation, redness and pain. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Eye Contact:
Vapors can cause eye irritation. Contact can produce pain, inflammation and temporal eye damage.
Chronic Exposure:
Can cause headache, mental confusion, depression, liver effects, kidney effects, bronchitis, loss of appetite, nausea, lack of balance, and visual disturbances. Can cause dermatitis upon prolonged skin contact. Methylene chloride may cause cancer in humans.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders, eye problems, impaired liver, kidney, respiratory or cardiovascular function may be more susceptible to the effects of this substance.

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4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.
Ingestion:
If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:
Immediately flush skin with plenty of soap and water for at least 15 minutes while removing contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.
Eye Contact:

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Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

---

5. Fire Fighting Measures

Fire:
Autoignition temperature: 556C (1033F)
Flammable limits in air % by volume:
lel: 12; uel: 23
Forms flammable vapor-air mixtures above 100C (212F).
Explosion:
Concentrated can be ignited by a high intensity ignition source. Vapor may form flammable mixture in atmosphere that contains a high percentage of oxygen. Sealed containers may rupture when heated.
Fire Extinguishing Media:
Dry chemical, foam or carbon dioxide. Water spray may be used to keep fire exposed containers cool.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Combustion by-products include phosgene and hydrogen chloride gases. Structural firefighters' clothing provides only limited protection to the combustion products of this material.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

---

7. Handling and Storage

Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect against physical damage. Isolate from any source of heat or ignition. Outside or detached storage is recommended. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product. To minimize decomposition, all storage containers should be galvanized or lined with a phenolic coating. This material may corrode plastic and rubber. Wear special protective equipment (Sec. 8) for maintenance break-in or where exposures may exceed established exposure levels. Wash hands, face, forearms and neck when exiting restricted areas. Shower, dispose of outer clothing, change to clean garments at the end of the day. Avoid cross-contamination of street clothes. Wash hands before eating and do not eat, drink, or smoke in workplace. Odor Threshold: 205 - 307 ppm. The odor threshold only serves as a warning of exposure; not smelling it does not mean you are not being exposed.

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8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
Methylene Chloride (Dichloromethane):
- OSHA Permissible Exposure Limit (PEL) -
25 ppm (TWA), 125 ppm (STEL), 12.5 ppm (8-hour TWA - Action Level)
- ACGIH Threshold Limit Value (TLV) -
50 ppm (TWA), A3 - suspected human carcinogen.

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. The cartridges recommended for this material have a predicted service of less than 30 minutes at concentrations of ten times (10x) the exposure limits. Actual service life will vary considerbly, depending on concentration levels, temperature, humidity, and work rate. This substance has poor warning properties.

Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact. Neoprene is a recommended material for personal protective equipment. Natural rubber and polyvinyl chloride ARE NOT recommended materials for personal protective equipment.

Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

Other Control Measures:
Do not use closed circuit rebreathing system employing soda lime or other carbon dioxide absorber because of formation of toxic compounds capable of producing cranial nerve paralysis. See OSHA Standard for medical surveillance, record keeping, and reporting requirements for methylene chloride (29 CFR 1910.1052).

---

9. Physical and Chemical Properties

Appearance:
Clear, colorless liquid.

Odor:
Chloroform-like odor.

Solubility:
1.32 gm/100 gm water @ 20C.

Specific Gravity:
1.318 @ 25C

pH:
No information found.

% Volatiles by volume @ 21C (70F):
100

Boiling Point:
39.8C (104F)

Melting Point:
-97C (-143F)

Vapor Density (Air=1):
2.9

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Vapor Pressure (mm Hg):
350 @ 20C (68F)
Evaporation Rate (BuAc=1):
27.5

---

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products:
Emits highly toxic fumes of phosgene when heated to decomposition. Decomposes in a flame or hot surface to form toxic gas phosgene and corrosive mists of hydrochloric acid. Carbon dioxide and carbon monoxide may form when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Strong oxidizers, strong caustics, plastics, rubber, nitric acid, water + heat, and chemically active metals, such as aluminum and magnesium powder, sodium, potassium, and lithium. Avoid contact with open flames and electrical arcs. Liquid methylene chloride will attack some forms of plastics, rubber, and coatings.
Conditions to Avoid:
Moisture, heat, flames, ignition sources and incompatibles.

---

11. Toxicological Information

Toxicological Data:
Dichloromethane: Oral rat LD50: 1600 mg/kg; inhalation rat LC50: 52 gm/m3; investigated as a tumorigen, mutagen, reproductive effector.
Reproductive Toxicity:
Dichloromethane has been linked to spontaneous abortions in humans.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Methylene Chloride (75-09-2)	No	Yes	2B

---

12. Ecological Information

Environmental Fate:
When released into the soil, this material may leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material may biodegrade to a moderate extent. When released to water, this material is expected to quickly evaporate. This material has a log octanol-water partition coefficient of less than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material may be moderately degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material is expected to have a half-life of greater than 30 days. When released into the air, this material may be removed from the atmosphere to a moderate extent by wet deposition.
Environmental Toxicity:
The LC50/96-hour values for fish are over 100 mg/l. This material is not expected to be toxic to aquatic life.

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13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

---

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: RQ, DICHLOROMETHANE
Hazard Class: 6.1
UN/NA: UN1593
Packing Group: III
Information reported for product/size: 1000L

International (Water, I.M.O.)

Proper Shipping Name: RQ, DICHLOROMETHANE
Hazard Class: 6.1
UN/NA: UN1593
Packing Group: III
Information reported for product/size: 1000L

International (Air, I.C.A.O.)

Proper Shipping Name: RQ, DICHLOROMETHANE
Hazard Class: 6.1
UN/NA: UN1593
Packing Group: III
Information reported for product/size: 1000L

---

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Methylene Chloride (75-09-2)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Methylene Chloride (75-09-2)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\----------------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Methylene Chloride (75-09-2)	No	No	Yes	No

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Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Methylene Chloride (75-09-2)	1000	U080	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: No Pressure: No
Reactivity: No (Pure / Liquid)

WARNING:
THIS PRODUCT CONTAINS A CHEMICAL(S) KNOWN TO THE STATE OF CALIFORNIA TO CAUSE CANCER.

Australian Hazchem Code: 2Z
Poison Schedule: S5
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **2** Flammability: **1** Reactivity: **0**
Label Hazard Warning:
WARNING! HARMFUL IF SWALLOWED, INHALED OR ABSORBED THROUGH SKIN. AFFECTS CENTRAL NERVOUS SYSTEM, LIVER, CARDIOVASCULAR SYSTEM, AND BLOOD. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. SUSPECT CANCER HAZARD. MAY CAUSE CANCER. Risk of cancer depends on level and duration of exposure.
Label Precautions:
Do not breathe vapor.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Keep away from heat and flame.
Do not get in eyes, on skin, or on clothing.
Label First Aid:
If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. In all cases, get medical attention.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 14.
Disclaimer:
****
Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER

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Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

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MSDS Number: **A0446** * * * * * *Effective Date:* ***05/20/04*** * * * * * *Supercedes:* ***02/12/04***

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS | J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

ACETONE

1. Product Identification

Synonyms: Dimethylketone; 2-propanone; dimethylketal
CAS No.: 67-64-1
Molecular Weight: 58.08
Chemical Formula: (CH3)2CO
Product Codes:
J.T. Baker: 5008, 5018, 5356, 5580, 9001, 9002, 9003, 9004, 9005, 9006, 9007, 9008, 9009, 9010, 9015, 9024, 9036, 9125, 9254, 9271, A134, V655
Mallinckrodt: 0018, 2432, 2435, 2437, 2438, 2440, 2443, 2445, 2850, H451, H580, H981

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Acetone	67-64-1	99 - 100%	Yes

3. Hazards Identification

Emergency Overview

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL IF SWALLOWED OR INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM.

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SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 2 - Moderate
Flammability Rating: 3 - Severe (Flammable)
Reactivity Rating: 0 - None
Contact Rating: 3 - Severe
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

Inhalation:
Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache. Higher concentrations can produce central nervous system depression, narcosis, and unconsciousness.
Ingestion:
Swallowing small amounts is not likely to produce harmful effects. Ingestion of larger amounts may produce abdominal pain, nausea and vomiting. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms are expected to parallel inhalation.
Skin Contact:
Irritating due to defatting action on skin. Causes redness, pain, drying and cracking of the skin.
Eye Contact:
Vapors are irritating to the eyes. Splashes may cause severe irritation, with stinging, tearing, redness and pain.
Chronic Exposure:
Prolonged or repeated skin contact may produce severe irritation or dermatitis.
Aggravation of Pre-existing Conditions:
Use of alcoholic beverages enhances toxic effects. Exposure may increase the toxic potential of chlorinated hydrocarbons, such as chloroform, trichloroethane.

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4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.
Ingestion:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately.
Skin Contact:
Immediately flush skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting upper and lower eyelids occasionally. Get medical attention.

---

5. Fire Fighting Measures

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Fire:
Flash point: -20C (-4F) CC
Autoignition temperature: 465C (869F)
Flammable limits in air % by volume:
lel: 2.5; uel: 12.8
Extremely Flammable Liquid and Vapor! Vapor may cause flash fire.

Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Vapors can flow along surfaces to distant ignition source and flash back. Contact with strong oxidizers may cause fire. Sealed containers may rupture when heated. This material may produce a floating fire hazard. Sensitive to static discharge.

Fire Extinguishing Media:
Dry chemical, alcohol foam or carbon dioxide. Water may be ineffective. Water spray may be used to keep fire exposed containers cool, dilute spills to nonflammable mixtures, protect personnel attempting to stop leak and disperse vapors.

Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB® solvent adsorbent is recommended for spills of this product.

---

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
Acetone:
-OSHA Permissible Exposure Limit (PEL):
1000 ppm (TWA)

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-ACGIH Threshold Limit Value (TLV):
500 ppm (TWA), 750 ppm (STEL) A4 - not classifiable as a human carcinogen

Ventilation System:

A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

Personal Respirators (NIOSH Approved):

If the exposure limit is exceeded and engineering controls are not feasible, a half-face organic vapor respirator may be worn for up to ten times the exposure limit, or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. A full-face piece organic vapor respirator may be worn up to 50 times the exposure limit, or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. For emergencies or instances where the exposure levels are not known, use a full-face piece positive-pressure, air-supplied respirator. WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.

Skin Protection:

Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Eye Protection:

Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

Appearance:
Clear, colorless, volatile liquid.

Odor:
Fragrant, mint-like

Solubility:
Miscible in all proportions in water.

Specific Gravity:
0.79 @ 20C/4C

pH:
No information found.

% Volatiles by volume @ 21C (70F):
100

Boiling Point:
56.5C (133F) @ 760 mm Hg

Melting Point:
-95C (-139F)

Vapor Density (Air=1):
2.0

Vapor Pressure (mm Hg):
400 @ 39.5C (104F)

Evaporation Rate (BuAc=1):
ca. 7.7

---

10. Stability and Reactivity

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Stability:
Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products:
Carbon dioxide and carbon monoxide may form when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Concentrated nitric and sulfuric acid mixtures, oxidizing materials, chloroform, alkalis, chlorine compounds, acids, potassium t-butoxide.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

---

11. Toxicological Information

Oral rat LD50: 5800 mg/kg; Inhalation rat LC50: 50,100mg/m3; Irritation eye rabbit, Standard Draize, 20 mg severe; investigated as a tumorigen, mutagen, reproductive effector.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Acetone (67-64-1)	No	No	None

---

12. Ecological Information

Environmental Fate:
When released into the soil, this material is expected to readily biodegrade. When released into the soil, this material is expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material is expected to readily biodegrade. When released to water, this material is expected to quickly evaporate. This material has a log octanol-water partition coefficient of less than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material may be moderately degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material may be moderately degraded by photolysis. When released into the air, this material is expected to be readily removed from the atmosphere by wet deposition.
Environmental Toxicity:
This material is not expected to be toxic to aquatic life. The LC50/96-hour values for fish are over 100 mg/l.

---

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

---

14. Transport Information

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Domestic (Land, D.O.T.)

Proper Shipping Name: ACETONE
Hazard Class: 3
UN/NA: UN1090
Packing Group: II
Information reported for product/size: 188L

International (Water, I.M.O.)

Proper Shipping Name: ACETONE
Hazard Class: 3
UN/NA: UN1090
Packing Group: II
Information reported for product/size: 188L

---

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Acetone (67-64-1)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

		--Canada--		
Ingredient	Korea	DSL	NDSL	Phil.
Acetone (67-64-1)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\----------------

	-SARA 302-		------SARA 313------	
Ingredient	RQ	TPQ	List	Chemical Catg.
Acetone (67-64-1)	No	No	Yes	No

--------\Federal, State & International Regulations - Part 2\----------------

		-RCRA-	-TSCA-
Ingredient	CERCLA	261.33	8(d)
Acetone (67-64-1)	5000	U002	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: Yes
SARA 311/312: Acute: Yes Chronic: No Fire: Yes Pressure: No
Reactivity: No (Pure / Liquid)

Australian Hazchem Code: 2[Y]E
Poison Schedule: None allocated.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

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16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**

Label Hazard Warning:

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL IF SWALLOWED OR INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM.

Label Precautions:

Keep away from heat, sparks and flame.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor.
Avoid contact with eyes, skin and clothing.

Label First Aid:

Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. In all cases, get medical attention.

Product Use:

Laboratory Reagent.

Revision Information:

No Changes.

Disclaimer:

****

Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.

****

Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

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Test Codes
Method: 3545
Date
Analyst
Batch #

MDEQ-Environmental Lab
ASE-Solids Extraction Logbook
Instrument # 17

Surrogate #
Spike #

ATTACHMENT 22.3

		Diatomateous Earth #		NaSO4 #		Acetone #		hexane #		MeCl #	
Vial	Cell	Sample ID #	Fraction #	Collection Date	Sample Weight (grams)	% Total Solids	Surrogate Volume (mL)	Spike Volume (mL)	Final Volume (mL)	Test Code	Comments
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
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23											
24											

COMMENTS

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Attachment 22.4

MDEQ-ENVIRONMENTAL LAB
STOCK TRACKING LOG

Environmental Lab Tracking Number	QTY (each)	Filley Tracking Number	Date Received (at lab)	Receiver Initials	Chemical,Analyte, Reagent or Solvent	Vendor and Catalog #	PO #	Manufacturer and Lot Number	Expiration Date
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									

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Attachment 22.5

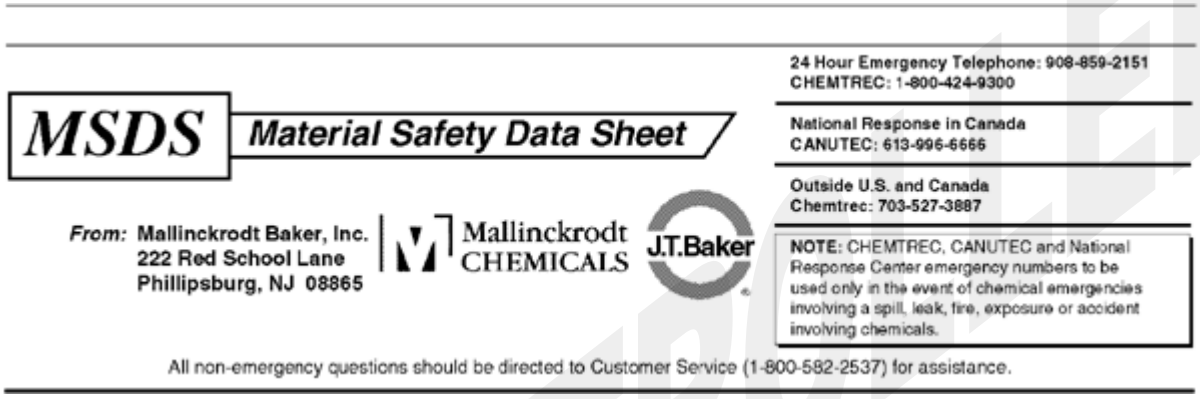
MDEQ-ENVIRONMENTAL LAB
SEMI-VOLATILE ORGANIC STANDARDS

SVOC Tracking Number	Date Prepared	EL or SVOC Tracking Number	Analyte or Mix	Preparers Initials	Solvent	Solvent Tracking Number	Initial Concentration*	Initial Volume/Wt Standard*	Final Volume*	Final Concentration*	Expiration Date
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											

UNCONTROLLED DOCUMENT

*INCLUDE UNITS

MSDS Number: **M2015** * * * * * *Effective Date:* ***08/10/04*** * * * * * *Supercedes:* ***11/12/01***



METHYL ALCOHOL

1. Product Identification

Synonyms: Wood alcohol; methanol; carbinol
CAS No.: 67-56-1
Molecular Weight: 32.04
Chemical Formula: CH3OH
Product Codes:
J.T. Baker: 5217, 5370, 5595, 5794, 5811, 5842, 5869, 9049, 9063, 9065, 9066, 9067, 9069, 9070, 9071, 9073, 9075, 9076, 9077, 9091, 9093, 9096, 9097, 9098, 9263, 9822, 9830, V654
Mallinckrodt: 3004, 3006, 3016, 3017, 3018, 3024, 3041, 3701, 4295, 5160, 8814, H080, H488, H603, H985, V079, V571

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Methyl Alcohol	67-56-1	100%	Yes

3. Hazards Identification

Emergency Overview

POISON! DANGER! VAPOR HARMFUL. MAY BE FATAL OR CAUSE BLINDNESS IF SWALLOWED. HARMFUL IF INHALED OR ABSORBED THROUGH SKIN. CANNOT BE MADE NONPOISONOUS.

UNCONTROLLED DOCUMENT

FLAMMABLE LIQUID AND VAPOR. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM AND LIVER.

SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Poison)
Flammability Rating: 3 - Severe (Flammable)
Reactivity Rating: 1 - Slight
Contact Rating: 3 - Severe (Life)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

Inhalation:
A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Once absorbed into the body, it is very slowly eliminated. Symptoms of overexposure may include headache, drowsiness, nausea, vomiting, blurred vision, blindness, coma, and death. A person may get better but then worse again up to 30 hours later.
Ingestion:
Toxic. Symptoms parallel inhalation. Can intoxicate and cause blindness. Usual fatal dose: 100-125 milliliters.
Skin Contact:
Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure.
Eye Contact:
Irritant. Continued exposure may cause eye lesions.
Chronic Exposure:
Marked impairment of vision has been reported. Repeated or prolonged exposure may cause skin irritation.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired liver or kidney function may be more susceptible to the effects of the substance.

---

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.
Ingestion:
Induce vomiting immediately as directed by medical personnel. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:
Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

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5. Fire Fighting Measures

Fire:
Flash point: 12C (54F) CC
Autoignition temperature: 464C (867F)
Flammable limits in air % by volume:
lel: 6.0; uel: 36
Flammable Liquid and Vapor!
Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Moderate explosion hazard and dangerous fire hazard when exposed to heat, sparks or flames. Sensitive to static discharge.
Fire Extinguishing Media:
Use alcohol foam, dry chemical or carbon dioxide. (Water may be ineffective.)
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Use water spray to blanket fire, cool fire exposed containers, and to flush non-ignited spills or vapors away from fire. Vapors can flow along surfaces to distant ignition source and flash back.

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6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB® solvent adsorbent is recommended for spills of this product.

---

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product. Do Not attempt to clean empty containers since residue is difficult to remove. Do not pressurize, cut, weld, braze, solder, drill, grind or expose such containers to heat, sparks, flame, static electricity or other sources of ignition: they may explode and cause injury or death.

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8. Exposure Controls/Personal Protection

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Airborne Exposure Limits:
For Methyl Alcohol:
- OSHA Permissible Exposure Limit (PEL):
200 ppm (TWA)
- ACGIH Threshold Limit Value (TLV):
200 ppm (TWA), 250 ppm (STEL) skin
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details. Use explosion-proof equipment.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded and engineering controls are not feasible, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. Breathing air quality must meet the requirements of the OSHA respiratory protection standard (29CFR1910.134). This substance has poor warning properties.
Skin Protection:
Rubber or neoprene gloves and additional protection including impervious boots, apron, or coveralls, as needed in areas of unusual exposure.
Eye Protection:
Use chemical safety goggles. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

Appearance:
Clear, colorless liquid.
Odor:
Characteristic odor.
Solubility:
Miscible in water.
Specific Gravity:
0.8
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100
Boiling Point:
64.5C (147F)
Melting Point:
-98C (-144F)
Vapor Density (Air=1):
1.1
Vapor Pressure (mm Hg):
97 @ 20C (68F)
Evaporation Rate (BuAc=1):
5.9

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10. Stability and Reactivity

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Stability:
Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products:
May form carbon dioxide, carbon monoxide, and formaldehyde when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Strong oxidizing agents such as nitrates, perchlorates or sulfuric acid. Will attack some forms of plastics, rubber, and coatings. May react with metallic aluminum and generate hydrogen gas.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

---

11. Toxicological Information

Methyl Alcohol (Methanol) Oral rat LD50: 5628 mg/kg; inhalation rat LC50: 64000 ppm/4H; skin rabbit LD50: 15800 mg/kg; Irritation data-standard Draize test: skin, rabbit: 20mg/24 hr. Moderate; eye, rabbit: 100 mg/24 hr. Moderate. Investigated as a mutagen, reproductive effector.

--------\Cancer Lists\----------

	---NTP Carcinogen---		
Ingredient	Known	Anticipated	IARC Category
Methyl Alcohol (67-56-1)	No	No	None

---

12. Ecological Information

Environmental Fate:
When released into the soil, this material is expected to readily biodegrade. When released into the soil, this material is expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into the water, this material is expected to have a half-life between 1 and 10 days. When released into water, this material is expected to readily biodegrade. When released into the air, this material is expected to exist in the aerosol phase with a short half-life. When released into the air, this material is expected to be readily degraded by reaction with photochemically produced hydroxyl radicals. When released into air, this material is expected to have a half-life between 10 and 30 days. When released into the air, this material is expected to be readily removed from the atmosphere by wet deposition.
Environmental Toxicity:
This material is expected to be slightly toxic to aquatic life.

---

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

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14. Transport Information

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Domestic (Land, D.O.T.)

Proper Shipping Name: METHANOL
Hazard Class: 3
UN/NA: UN1230
Packing Group: II
Information reported for product/size: 358LB

International (Water, I.M.O.)

Proper Shipping Name: METHANOL
Hazard Class: 3, 6.1
UN/NA: UN1230
Packing Group: II
Information reported for product/size: 358LB

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15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Methyl Alcohol (67-56-1)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Methyl Alcohol (67-56-1)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\----------------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Methyl Alcohol (67-56-1)	No	No	Yes	No

--------\Federal, State & International Regulations - Part 2\----------------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Methyl Alcohol (67-56-1)	5000	U154	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: Yes Pressure: No
Reactivity: No (Pure / Liquid)

Australian Hazchem Code: 2PE
Poison Schedule: S6
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

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16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**

Label Hazard Warning:

POISON! DANGER! VAPOR HARMFUL. MAY BE FATAL OR CAUSE BLINDNESS IF SWALLOWED. HARMFUL IF INHALED OR ABSORBED THROUGH SKIN. CANNOT BE MADE NONPOISONOUS. FLAMMABLE LIQUID AND VAPOR. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM AND LIVER.

Label Precautions:

Avoid breathing vapor.
Avoid contact with eyes, skin and clothing.
Wash thoroughly after handling.
Keep container closed.
Use only with adequate ventilation.
Keep away from heat, sparks and flame.

Label First Aid:

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. If swallowed, induce vomiting immediately as directed by medical personnel. Never give anything by mouth to an unconscious person. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. In all cases get medical attention immediately.

Product Use:

Laboratory Reagent.

Revision Information:

No Changes.

Disclaimer:

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Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.

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Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

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SEDIMENT PREPARATION (%TOTAL SOLID & DRYING)

Oven Temperature
Initial Temp. (105 ± 5°C) _____
Final Temp. (105 ± 5°C) _____

Calibration Weights
Initial 20 g _____ Initial 50 g _____
Final 20 g _____ Final 50 g _____
Balance Centered Y N

Run # _____
Date _____
Analyst _____

WORK ORDER #	MS	OS	DRY & GRIND	TARE WT.	WET WT.	DRY WT	%TS	COMMENTS
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MSDS Number: **H2381** * * * * * *Effective Date:* ***08/10/04*** * * * * * *Supercedes:* ***11/02/01***

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

J.T.Baker®

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. And Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

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HEXANE

1. Product Identification

Synonyms: Hexanes,Normal Hexane; Hexyl Hydride; Hexane 95%
CAS No.: 110-54-3 (n-hexane)
Molecular Weight: 86.18
Chemical Formula: CH3(CH2)4CH3 n-hexane
Product Codes: 9262, 9304, 9308, N168

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Hexane	110-54-3	85 - 100%	Yes
Methylcyclopentane	96-37-7	1 - 2%	Yes
Trace amount of Benzene (10 ppm)	071-43-2	*	No

3. Hazards Identification

Emergency Overview

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 2 - Moderate
Flammability Rating: 3 - Severe (Flammable)
Reactivity Rating: 0 - None
Contact Rating: 2 - Moderate
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

The health hazards addressed are for the major component: n-hexane.

Inhalation:
Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Greater exposure may cause muscle weakness, numbness of the extremities, unconsciousness and death.
Ingestion:
May produce abdominal pain, nausea. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms expected to parallel inhalation.
Skin Contact:
May cause redness, irritation, with dryness, cracking.
Eye Contact:
Vapors may cause irritation. Splashes may cause redness and pain.
Chronic Exposure:
Repeated or prolonged skin contact may defat the skin and produce irritation and dermatitis. Chronic inhalation may cause peripheral nerve disorders and central nervous system effects.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance. May affect the developing fetus.

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4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:
Aspiration hazard. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:
Remove any contaminated clothing. Wipe off excess from skin. Wash skin with soap and water for at least 15 minutes. Get medical attention if irritation develops or persists.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

Note to Physician:
BEI=2,5-hexadione in urine, sample at end of shift at workweeks end, 5 mg/g creatine. Also, measure n-hexane in

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expired air. Analgesics may be necessary for pain management, there is no specific antidote. Monitor arterial blood gases in cases of severe aspiration.

---

5. Fire Fighting Measures

Fire:
Flash point: -23C (-9F) CC
Autoignition temperature: 224C (435F)
Flammable limits in air % by volume:
lel: 1.2; uel: 7.7
Extremely Flammable Liquid and Vapor! Vapor may cause flash fire. Dangerous fire hazard when exposed to heat or flame.
Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Contact with oxidizing materials may cause extremely violent combustion.Explodes when mixed @ 28C with dinitrogen tetraoxide. Sensitive to static discharge.
Fire Extinguishing Media:
Dry chemical, foam or carbon dioxide. Water may be ineffective.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Water spray may be used to keep fire exposed containers cool. Vapors can flow along surfaces to distant ignition source and flash back. Vapor explosion hazard exists indoors, outdoors, or in sewers.

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6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB® solvent adsorbent is recommended for spills of this product.

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7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from direct sunlight and any area where the fire hazard may be acute. Store in tightly closed containers (preferably under nitrogen atmosphere). Outside or detached storage is preferred. Inside storage should be in a standard flammable liquids storage room or cabinet. Separate from oxidizing materials. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

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8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
N-Hexane [110-54-3]:
-OSHA Permissible Exposure Limit (PEL): 500 ppm (TWA)
-ACGIH Threshold Limit Value (TLV): 50 ppm (TWA), Skin
other isomers of hexane
-ACGIH Threshold Limit Value (TLV): 500 ppm (TWA),1000ppm (STEL)
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded and engineering controls are not feasible, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. Breathing air quality must meet the requirements of the OSHA respiratory protection standard (29CFR1910.134).
Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.
Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

Appearance:
Clear, colorless liquid.
Odor:
Light odor.
Solubility:
Insoluble in water.
Specific Gravity:
0.66
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100
Boiling Point:
ca. 68C (ca. 154F)
Melting Point:
ca. -95C (ca. -139F)
Vapor Density (Air=1):
3.0
Vapor Pressure (mm Hg):
130 @ 20C (68F)
Evaporation Rate (BuAc=1):
9

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10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Heat will contribute to instability.
Hazardous Decomposition Products:
May produce acrid smoke and irritating fumes when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Strong oxidizers.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

11. Toxicological Information

N-Hexane: Oral rat LD50: 28710 mg/kg. Irritation eye rabbit: 10 mg mild. Investigated as a tumorigen, mutagen and reproductive effector.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Hexane (110-54-3)	No	No	None
Methylcyclopentane (96-37-7)	No	No	None
Trace amount of Benzene (10 ppm) (071-43-2)	Yes	No	1

12. Ecological Information

Environmental Fate:
When released into the soil, this material may biodegrade to a moderate extent. When released into the soil, this material is not expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material may biodegrade to a moderate extent. When released to water, this material is expected to quickly evaporate. When released into the water, this material is expected to have a half-life between 1 and 10 days. This material has an estimated bioconcentration factor (BCF) of less than 100. This material has a log octanol-water partition coefficient of greater than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material is expected to be readily degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material is expected to have a half-life between 1 and 10 days.
Environmental Toxicity:
No information found.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product

UNCONTROLLED DOCUMENT

may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: HEXANES
Hazard Class: 3
UN/NA: UN1208
Packing Group: II
Information reported for product/size: 215L

International (Water, I.M.O.)

Proper Shipping Name: HEXANES
Hazard Class: 3
UN/NA: UN1208
Packing Group: II
Information reported for product/size: 215L

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Hexane (110-54-3)	Yes	Yes	Yes	Yes
Methylcyclopentane (96-37-7)	Yes	Yes	No	Yes
Trace amount of Benzene (10 ppm) (071-43-2)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Hexane (110-54-3)	Yes	Yes	No	Yes
Methylcyclopentane (96-37-7)	Yes	Yes	No	Yes
Trace amount of Benzene (10 ppm) (071-43-2)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\----------------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Hexane (110-54-3)	No	No	Yes	No
Methylcyclopentane (96-37-7)	No	No	No	No
Trace amount of Benzene (10 ppm) (071-43-2)	No	No	Yes	No

--------\Federal, State & International Regulations - Part 2\----------------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Hexane (110-54-3)	5000	No	No
Methylcyclopentane (96-37-7)	No	No	No
Trace amount of Benzene (10 ppm)	10	U019	No

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Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: Yes Pressure: No
Reactivity: No (Mixture / Liquid)

WARNING:
THIS PRODUCT CONTAINS A CHEMICAL(S) KNOWN TO THE STATE OF CALIFORNIA TO CAUSE CANCER.

Australian Hazchem Code: 3[Y]E
Poison Schedule: None allocated.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

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16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.
Label Precautions:
Keep away from heat, sparks and flame.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor or mist.
Avoid contact with eyes, skin and clothing.
Label First Aid:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. In all cases call a physician.
Product Use:
Laboratory Reagent.
Revision Information:
No Changes.
Disclaimer:
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Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

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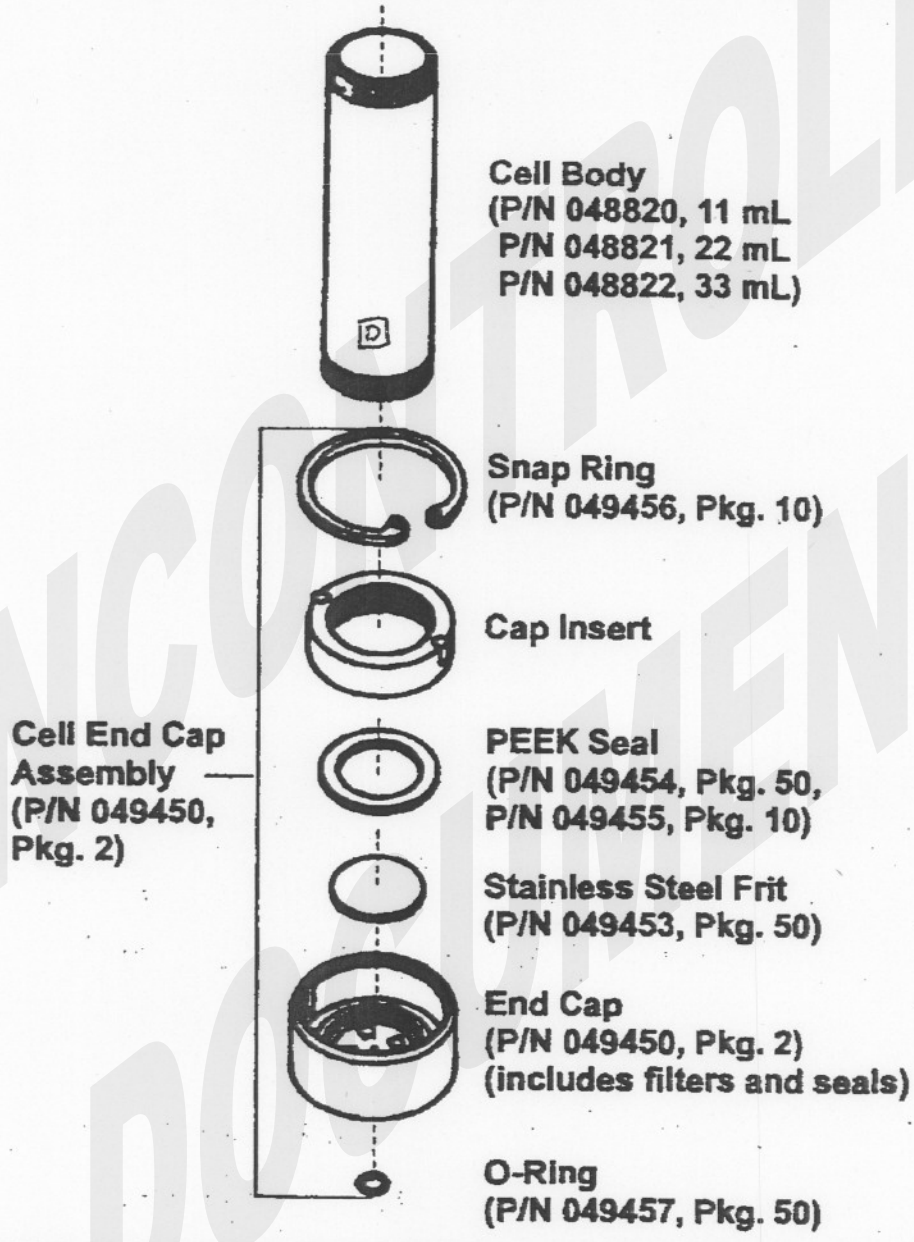

Figure 5-1. Cell Assembly

ıe stainless steel frit from the bottom of the end cap.
frit by sonicating in solvent, or replace it.

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To fill the cell:

1. Dionex recommends inserting a disposable cellulose filter (P/N 049458, box of 100) in the bottom of the cell before filling. The cellulose filter prevents blockage of the bottom cap's stainless steel frit.

IMPORTANT **Always hand-tighten the bottom cell cap onto the cell body before installing the cellulose filter. Do not place the filter in the bottom cap and then install the cap, because this creates an improper seal and causes leaks.**

To insert the filter, unscrew the top cap from the cell body. Push the filter into the cell bottom with the tool (P/N 049495) provided in the Ship Kit (see Figure 3-2).

IMPORTANT **Hand-tighten the cell caps only. The use of a wrench or other tool can damage the cell.**

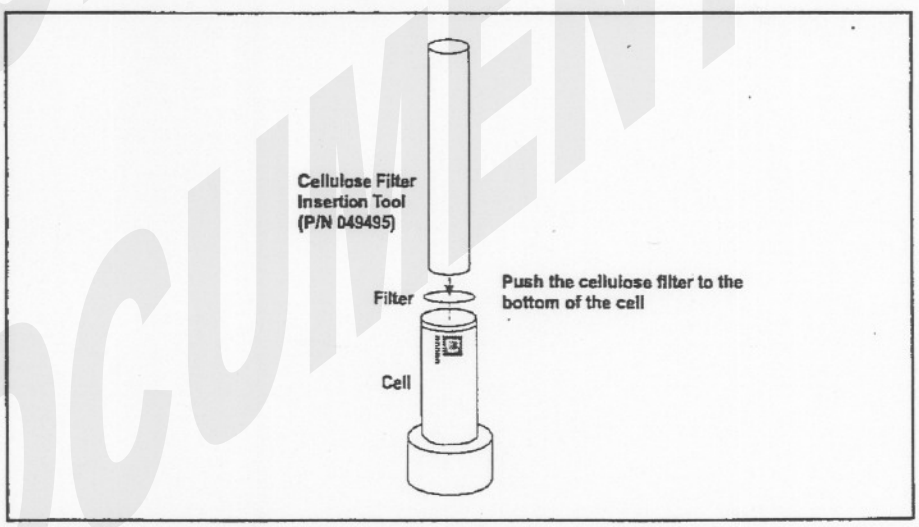

Figure 3-2. Cellulose Filter Installation

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MSDS Number: **P3973** * * * * * *Effective Date:* ***01/03/06*** * * * * * *Supercedes:* ***08/31/04***

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

PHOSPHORIC ACID

1. Product Identification

Synonyms: Ortho-phosphoric acid; white phosphoric acid
CAS No.: 7664-38-2
Molecular Weight: 98.00
Chemical Formula: H3PO4 in H2O
Product Codes:
J.T. Baker: 0238, 0248, 0259, 0260, 0262, 0263, 0264, 0268, 0273, 0274, 5372, 5682, 5686, 5804, 5841, 5854, 6908
Mallinckrodt: 2779, 2788, 2796, 3563, H106, XM-223

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Phosphoric Acid	7664-38-2	55 - 95%	Yes
Water	7732-18-5	5 - 45%	No

3. Hazards Identification

Emergency Overview

DANGER! CORROSIVE. CAUSES SEVERE IRRITATION AND BURNS TO EVERY AREA OF CONTACT. HARMFUL IF SWALLOWED OR INHALED.

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SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe
Flammability Rating: 0 - None
Reactivity Rating: 2 - Moderate
Contact Rating: 4 - Extreme (Corrosive)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: White (Corrosive)

Potential Health Effects

Inhalation:
Inhalation is not an expected hazard unless misted or heated to high temperatures. Mist or vapor inhalation can cause irritation to the nose, throat, and upper respiratory tract. Severe exposures can lead to a chemical pneumonitis.
Ingestion:
Corrosive. May cause sore throat, abdominal pain, nausea, and severe burns of the mouth, throat, and stomach. Severe exposures can lead to shock, circulatory collapse, and death.
Skin Contact:
Corrosive. May cause redness, pain, and severe skin burns.
Eye Contact:
Corrosive. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Chronic Exposure:
No information found.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems, or impaired respiratory function may be more susceptible to the effects of the substance.

---

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician immediately.
Ingestion:
If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:
Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Call a physician, immediately. Wash clothing before reuse.
Eye Contact:
Immediately flush eyes with gentle but large stream of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Call a physician immediately.

---

5. Fire Fighting Measures

Fire:
Not considered to be a fire hazard. Contact with most metals causes formation of flammable and explosive hydrogen gas.

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Explosion:
Not considered to be an explosion hazard.
Fire Extinguishing Media:
Use any means suitable for extinguishing surrounding fire. Water spray may be used to keep fire exposed containers cool. If water is used, use in abundance to control heat and acid build-up.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Neutralize with alkaline material (soda ash, lime), then absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker NEUTRASORB® or TEAM® 'Low Na+' acid neutralizers are recommended for spills of this product.

---

7. Handling and Storage

Keep in a tightly closed container. Protect from physical damage. Store in a cool, dry, ventilated area away from sources of heat, moisture, incompatibilities, and direct sunlight. Corrosive to mild steel. Store in rubber lined or 316 stainless steel designed for phosphoric acid. Do not wash out container and use it for other purposes. When diluting, the acid should always be added slowly to water and in small amounts. Never use hot water and never add water to the acid. Water added to acid can cause uncontrolled boiling and splashing. Protect from freezing. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
-OSHA Permissible Exposure Limit (PEL):
1 mg/m3 (TWA)

-ACGIH Threshold Limit Value (TLV):
1 mg/m3 (TWA), 3 mg/m3 (STEL)
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, a full facepiece respirator with high efficiency dust/mist filter may be worn up to 50 times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator

supplier, whichever is lowest. For emergencies or instances where the exposure levels are not known, use a full-facepiece positive-pressure, air-supplied respirator. WARNING: Air purifying respirators do not protect workers in oxygen-deficient atmospheres.

Skin Protection:

Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Eye Protection:

Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

Physical data below refers to concentrated phosphoric acid.

Appearance:

Clear, colorless syrupy liquid.

Odor:

Odorless.

Solubility:

Miscible in all proportions in water.

Specific Gravity:

1.69 @ 25C

pH:

1.5 (0.1 N aqueous solution)

% Volatiles by volume @ 21C (70F):

100

Boiling Point:

158C (316F)

Melting Point:

21C (70F)

Vapor Density (Air=1):

3.4

Vapor Pressure (mm Hg):

0.03 @ 20C (68F)

Evaporation Rate (BuAc=1):

No information found.

---

10. Stability and Reactivity

Stability:

Stable under ordinary conditions of use and storage. Substance can supercool without crystallizing.

Hazardous Decomposition Products:

Phosphorus oxides may form when heated to decomposition.

Hazardous Polymerization:

Will not occur.

Incompatibilities:

Liberates explosive hydrogen gas when reacting with chlorides and stainless steel. Can react violently with sodium tetrahydroborate. Exothermic reactions with aldehydes, amines, amides, alcohols and glycols, azo-compounds, carbamates, esters, caustics, phenols and cresols, ketones, organophosphates, epoxides, explosives, combustible materials, unsaturated halides, and organic peroxides. phosphoric acid forms flammable gases with sulfides,

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mercaptans, cyanides and aldehydes. It also forms toxic fumes with cyanides, sulfide, fluorides, organic peroxides, and halogenated organics. Mixtures with nitromethane are explosive.
Conditions to Avoid:
Incompatibles.

---

11. Toxicological Information

Oral rat LD50: 1530 mg/kg; investigated as a mutagen.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Phosphoric Acid (7664-38-2)	No	No	None
Water (7732-18-5)	No	No	None

---

12. Ecological Information

Environmental Fate:
When released into the soil, this material may leach into groundwater. When released to water, acidity may be readily reduced by natural water hardness minerals. The phosphate, however, may persist indefinitely.
Environmental Toxicity:
No information found.

---

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

---

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: Phosphoric Acid Solution
Hazard Class: 8
UN/NA: UN1805
Packing Group: III
Information reported for product/size: 350LB

International (Water, I.M.O.)

Proper Shipping Name: Phosphoric Acid Solution
Hazard Class: 8
UN/NA: UN1805

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Packing Group: III
Information reported for product/size: 350LB

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Phosphoric Acid (7664-38-2)	Yes	Yes	Yes	Yes
Water (7732-18-5)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Phosphoric Acid (7664-38-2)	Yes	Yes	No	Yes
Water (7732-18-5)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\----------------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Phosphoric Acid (7664-38-2)	No	No	No	No
Water (7732-18-5)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\----------------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Phosphoric Acid (7664-38-2)	5000	No	No
Water (7732-18-5)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: No Fire: No Pressure: No
Reactivity: No (Pure / Liquid)

Australian Hazchem Code: 2R
Poison Schedule: S5
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

16. Other Information

NFPA Ratings: Health: **3** Flammability: **0** Reactivity: **0**
Label Hazard Warning:
DANGER! CORROSIVE. CAUSES SEVERE IRRITATION AND BURNS TO EVERY AREA OF CONTACT. HARMFUL IF SWALLOWED OR INHALED.
Label Precautions:
Do not get in eyes, on skin, or on clothing.
Keep container closed.
Use only with adequate ventilation.

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Do not breathe vapor or mist.
Wash thoroughly after handling.

Label First Aid:

In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In all cases call a physician immediately.

Product Use:

Laboratory Reagent.

Revision Information:

MSDS Section(s) changed since last revision of document include: 14.

Disclaimer:

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Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

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MSDS Number: **S5018** * * * * * *Effective Date:* ***06/17/03*** * * * * * *Supercedes:* ***02/18/03***

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

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SODIUM SULFATE ANHYDROUS

1. Product Identification

Synonyms: Disodium sulfate; sodium sulfate anhydrous; sulfuric acid, disodium salt; sodium sulfate; Speed-dry
CAS No.: 7757-82-6
Molecular Weight: 142.04
Chemical Formula: Na2 SO4
Product Codes:
J.T. Baker: 3375, 3891, 3898, 3902, 8074
Mallinckrodt: 6775, 7803, 8016, 8020, 8022, 8024, 8025, 8026, 8028, 9675

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Sodium Sulfate	7757-82-6	90 - 100%	Yes

3. Hazards Identification

Emergency Overview

As part of good industrial and personal hygiene and safety procedure, avoid all unnecessary exposure to the chemical substance and ensure prompt removal from skin, eyes and clothing.

SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 1 - Slight
Flammability Rating: 1 - Slight
Reactivity Rating: 2 - Moderate
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT
Storage Color Code: Green (General Storage)

Potential Health Effects

Inhalation:
Not expected to be a health hazard.
Ingestion:
Mildly toxic by ingestion. Slowly absorbed from the alimentary tract. Because of osmotic activity, it will draw water into the lumen of the bowel and may cause purging, fluid loss, blood in stools, fall of blood pressure, and high sodium levels in the blood.
Skin Contact:
No adverse effects expected.
Eye Contact:
No adverse effects expected but dust may cause mechanical irritation.
Chronic Exposure:
No information found.
Aggravation of Pre-existing Conditions:
No information found.

---

4. First Aid Measures

Inhalation:
Not expected to require first aid measures.
Ingestion:
Give several glasses of water to drink to dilute. If large amounts were swallowed, get medical advice.
Skin Contact:
Wash exposed area with soap and water. Get medical advice if irritation develops.
Eye Contact:
Wash thoroughly with running water. Get medical advice if irritation develops.

---

5. Fire Fighting Measures

Fire:
Not considered to be a fire hazard.
Explosion:
Not considered to be an explosion hazard, but violent explosions occur when potassium sulfate and sodium sulfate are melted with aluminum.
Fire Extinguishing Media:
Use any means suitable for extinguishing surrounding fire.
Special Information:
Use protective clothing and breathing equipment appropriate for the surrounding fire.

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6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Spills: Sweep up and containerize for reclamation or disposal. Vacuuming or wet sweeping may be used to avoid dust dispersal.

7. Handling and Storage

Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect against physical damage. Isolate from incompatible substances. Containers of this material may be hazardous when empty since they retain product residues (dust, solids); observe all warnings and precautions listed for the product.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
None established.
Ventilation System:
In general, dilution ventilation is a satisfactory health hazard control for this substance. However, if conditions of use create discomfort to the worker, a local exhaust system should be considered.
Personal Respirators (NIOSH Approved):
For conditions of use where exposure to dust or mist is apparent and engineering controls are not feasible, a particulate respirator (NIOSH type N95 or better filters) may be worn. If oil particles (e.g. lubricants, cutting fluids, glycerine, etc.) are present, use a NIOSH type R or P filter. For emergencies or instances where the exposure levels are not known, use a full-face positive-pressure, air-supplied respirator. WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.
Skin Protection:
Wear protective gloves and clean body-covering clothing.
Eye Protection:
Use chemical safety goggles. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

Appearance:
Fine white crystalline granules.
Odor:
Odorless.
Solubility:
Soluble in approx. 3.6 parts water, 1 in 2 parts maximum @ 33C (91F).
Density:
2.68
pH:
No information found.

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% Volatiles by volume @ 21C (70F):
0
Boiling Point:
No information found.
Melting Point:
844C (1551F)
Vapor Density (Air=1):
No information found.
Vapor Pressure (mm Hg):
No information found.
Evaporation Rate (BuAc=1):
No information found.

---

10. Stability and Reactivity

Stability:
Stable in tightly closed containers under normal conditions of storage.
Hazardous Decomposition Products:
Oxides of sulfur and sodium may form when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
In combination with sodium sulfate, aluminum and magnesium will explode @ 800C (1472F); strong mineral acids and bases.
Conditions to Avoid:
Air, moisture, and incompatibles.

---

11. Toxicological Information

No LD50/LC50 information found relating to normal routes of occupational exposure. Investigated as a tumorigen, mutagen, reproductive effector.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Sodium Sulfate (7757-82-6)	No	No	None

---

12. Ecological Information

Environmental Fate:
When released into the soil, this material is expected to leach into groundwater. This material is not expected to significantly bioaccumulate.
Environmental Toxicity:
This material is not expected to be toxic to aquatic life. The LC50/96-hour values for fish are over 100 mg/l. The EC50/48-hour values for daphnia are over 100 mg/l.

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13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be managed in an appropriate and approved waste disposal facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

---

14. Transport Information

Not regulated.

---

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Sodium Sulfate (7757-82-6)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Sodium Sulfate (7757-82-6)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\----------------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Sodium Sulfate (7757-82-6)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\----------------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Sodium Sulfate (7757-82-6)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: No Chronic: No Fire: No Pressure: No
Reactivity: No (Pure / Solid)

Australian Hazchem Code: None allocated.
Poison Schedule: None allocated.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **1** Flammability: **0** Reactivity: **0**
Label Hazard Warning:

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As part of good industrial and personal hygiene and safety procedure, avoid all unnecessary exposure to the chemical substance and ensure prompt removal from skin, eyes and clothing.

Label Precautions:

None.

Label First Aid:

Not applicable.

Product Use:

Laboratory Reagent.

Revision Information:

MSDS Section(s) changed since last revision of document include: 3.

Disclaimer:

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Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.

****

Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

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MSDS Number: **D1288** * * * * * *Effective Date:* ***05/08/03*** * * * * * *Supercedes:* ***09/14/00***

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

J.T.Baker®

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300
National Response in Canada
CANUTEC: 613-996-6666
Outside U.S. And Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

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Diatomaceous Earth

1. Product Identification

Synonyms: Diatomite; Celite®; Kieselguhr Soda Ash Flux Calcined
CAS No.: 68855-54-9
Molecular Weight: Not applicable.
Chemical Formula: Not applicable.
Product Codes: 1939

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Kieselguhr, Soda Ash Flux-calcined	68855-54-9	100%	Yes
This product may contain up to 75% crystalline silica:			
Cristobalite	14464-46-1	< 70%	Yes
Quartz	14808-60-7	< 5%	Yes

3. Hazards Identification

Emergency Overview

WARNING! HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. MAY AFFECT LUNGS. CANCER HAZARD. CONTAINS CRYSTALLINE SILICA WHICH CAN CAUSE CANCER. Risk of cancer depends upon duration and level of exposure.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 4 - Extreme (Cancer Causing)
Flammability Rating: 0 - None
Reactivity Rating: 0 - None
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES
Storage Color Code: Blue (Health)

Potential Health Effects

Inhalation:
Causes dryness and irritation to the respiratory tract. Symptoms may include coughing, sore throat, breathing difficulty (dyspnea), and wheezing. Excessive inhalation may cause decreased pulmonary function, lung damage and silicosis. Acute silicosis is manifested by dyspnea, fever, cough and weight loss. Severe respiratory symptoms may lead to death.
Ingestion:
No adverse effects expected.
Skin Contact:
Causes irritation with dryness and abrasion.
Eye Contact:
Causes irritation, redness, and pain.
Chronic Exposure:
Prolonged inhalation exposure may produce silicosis. Chronic symptoms include cough, dyspnea, wheezing, increased susceptibility to tuberculosis, decreased chest expansion, and repeated nonspecific chest illnesses. Progressive respiratory and cardiopulmonary impairment may be fatal. Chronic inhalation of crystalline silica is a lung cancer hazard.
Aggravation of Pre-existing Conditions:
Persons with pre-existing respiratory or cardiopulmonary problems may be more susceptible to the effects of this substance.

---

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.
Ingestion:
Give several glasses of water to drink to dilute. If large amounts were swallowed, get medical advice.
Skin Contact:
Immediately flush skin with plenty of soap and water for at least 15 minutes. Remove contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

---

5. Fire Fighting Measures

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Fire:
Not considered to be a fire hazard.
Explosion:
Not considered to be an explosion hazard.
Fire Extinguishing Media:
Use any means suitable for extinguishing surrounding fire.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Keep unnecessary and unprotected people away from area of spill. Wear appropriate personal protective equipment as specified in Section 8. Spills: Pick up and place in a suitable container for reclamation or disposal, using a method that does not generate dust.

---

7. Handling and Storage

Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect against physical damage. When pouring into a container of flammable liquid, ground both containers electrically to prevent a static electric spark. Containers of this material may be hazardous when empty since they retain product residues (dust, solids); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
OSHA PERMISSIBLE LIMITS (PELs):
- For silica, amorphous, including natural diatomaceous earth (112926-00-8):
(80 mg/m3) / (%SiO2), (TWA).
- For silica, crystalline, quartz (14808-60-7):
(30mg/m3)/(%SiO2 + 2), (TWA), total dust;
(10 mg/m3)/(%SiO2 + 2), (TWA), respirable fraction;
where "%SiO2" is the percentage of crystalline silica
determined by airborne samples, as defined by
29 CFR 1910.1000, Z-3.
- For silica, crystalline, tridymite (15468-32-3) or cristobalite (14464-46-1):
Use one-half of the quartz exposure limits.

ACGIH THRESHOLD LIMIT VALUES:
- For silica gel & precipitated silica (11926-00-8):
10 mg/m3 (TWA).
- For diatomaceous earth, uncalcined (61790-53-2):
10 mg/m3 (TWA), inhalable particulate;
3 mg/m3 (TWA), respirable particulate;
(for particulate matter containing no asbestos and < 1% crystalline silica).
- For fused silica (60676-86-0):

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0.1 mg/m3 (TWA), respirable fraction.
- For silica, crystalline, quartz (14808-60-7):
0.05 mg/m3 (TWA), respirable fraction, A2 - Suspected Human Carcinogen.
- For silica, crystalline, tridymite (15468-32-3):
0.05 mg/m3 (TWA), respirable fraction.
- For silica, crystalline, cristobalite (14464-46-1):
0.05 mg/m3 (TWA), respirable fraction.
- For silica, crystalline, tripoli (1317-95-9):
0.1 mg/m3 of contained respirable quartz (TWA), respirable fraction.

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded and engineering controls are not feasible, a half-face high efficiency particulate respirator (NIOSH type N100 filter) may be worn for up to ten times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. A full-face piece high efficiency particulate respirator (NIOSH type N100 filter) may be worn up to 50 times the exposure limit, or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. If oil particles (e.g. lubricants, cutting fluids, glycerine, etc.) are present, use a NIOSH type R or P filter. For emergencies or instances where the exposure levels are not known, use a full-facepiece positive-pressure, air-supplied respirator. WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres. Where respirators are required, you must have a written program covering the basic requirements in the OSHA respirator standard. These include training, fit testing, medical approval, cleaning, maintenance, cartridge change schedules, etc. See 29CFR1910.134 for details.

Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Eye Protection:
Use chemical safety goggles and/or full face shield where dusting or splashing of solutions is possible. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

Appearance:
White to pink Crystalline solid.

Odor:
No information found.

Solubility:
Slight (0.1-1%)

Specific Gravity:
2.30

pH:
No information found.

% Volatiles by volume @ 21C (70F):
0

Boiling Point:
No information found.

Melting Point:

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No information found.
Vapor Density (Air=1):
Not applicable.
Vapor Pressure (mm Hg):
Not applicable.
Evaporation Rate (BuAc=1):
No information found.

---

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products:
No information found.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Reacts with hydrogen fluoride, fluorine, oxygen difluoride, chlorine trifluoride, strong acids, strong bases, and oxidizers.
Conditions to Avoid:
Moisture, extreme heat, and incompatibles.

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11. Toxicological Information

Toxicological Data:
No LD50/LC50 information found relating to normal routes of occupational exposure.

Silica, Amorphous:
- diatomaceous earth: investigated as a tumorigen.

Silica, Crystalline:
- tripoli: investigated as a tumorigen.
- tridymite: investigated as a tumorigen and mutagen.
- quartz: investigated as a tumorigen and mutagen.
- cristobalite: investigated as a tumorigen.
- fused: investigated as a tumorigen.

Carcinogenicity:
For Silica, Crystalline:
- Cristobalite (14464-46-1), quartz (14808-60-7), and tridymite (15468-32-3) are listed by NTP as known to be a human carcinogen.
- NIOSH considers cristobalite, tridymite, quartz, and tripoli (1317-95-9) to be potential occupational carcinogens.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Kieselguhr, Soda Ash Flux-calcined (68855-54-9)	No	No	None
Cristobalite (14464-46-1)	Yes	No	1

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Quartz (14808-60-7)	Yes	No	1

12. Ecological Information

Environmental Fate:
When released into the soil, this material is not expected to biodegrade. When released into water, this material is not expected to biodegrade.
Environmental Toxicity:
No information found.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be managed in an appropriate and approved waste disposal facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Not regulated.

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Kieselguhr, Soda Ash Flux-calcined (68855-54-9)	Yes	Yes	No	Yes
Cristobalite (14464-46-1)	Yes	Yes	Yes	Yes
Quartz (14808-60-7)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Kieselguhr, Soda Ash Flux-calcined (68855-54-9)	Yes	Yes	No	Yes
Cristobalite (14464-46-1)	Yes	Yes	No	Yes
Quartz (14808-60-7)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\--------

Ingredient	-SARA 302- RQ	TPQ	List	------SARA 313------ Chemical Catg.
Kieselguhr, Soda Ash Flux-calcined (68855-54-9)	No	No	No	No
Cristobalite (14464-46-1)	No	No	No	No
Quartz (14808-60-7)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\--------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)

Kieselguhr, Soda Ash Flux-calcined (68855-54-9)	No	No	No
Cristobalite (14464-46-1)	No	No	No
Quartz (14808-60-7)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: No Pressure: No
Reactivity: No (Pure / Solid)

WARNING:
THIS PRODUCT CONTAINS A CHEMICAL(S) KNOWN TO THE STATE OF CALIFORNIA TO CAUSE CANCER.

Australian Hazchem Code: None allocated.
Poison Schedule: None allocated.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

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16. Other Information

NFPA Ratings: Health: **2** Flammability: **0** Reactivity: **0**
Label Hazard Warning:
WARNING! HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. MAY AFFECT LUNGS. CANCER HAZARD. CONTAINS CRYSTALLINE SILICA WHICH CAN CAUSE CANCER. Risk of cancer depends upon duration and level of exposure.
Label Precautions:
Do not breathe dust.
Keep container closed.
Use only with adequate ventilation.
Do not get in eyes, on skin, or on clothing.
Wash thoroughly after handling.
Label First Aid:
If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. In all cases, get medical attention.
Product Use:
Laboratory Reagent.
Revision Information:
No Changes.
Disclaimer:
****

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Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

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Attachment 22.14

The Lancer dishwasher has two programs that we use. Program 5 uses deionized water and program 4 uses tap water.

Functions	Possibility	Our Program
PREWASH 1 PERIOD	0-30 MINUTES	1 MINUTE
FILLING	HOT/COLD	HOT
PREWASH 1 TEMPERATURE	0-95°C	50°C
PREWASH 1 DETERGENT	0-6 MINUTES	24 SECONDS
PREWASH 1 ADDITIVE 2 (ACID)	0-6 MINUTES	0
PREWASH 2 PERIOD	0-30 MINUTES	2 MINUTES
FILLING	HOT/COLD	HOT
PREWASH 2 TEMPERATURE	0-95°C	65°C
PREWASH 2 DETERGENT	0-6 MINUTES	24 SECONDS
PREWASH 2 ADDITIVE 2	0-6 MINUTES	0
PREWASH 3 PERIOD	0-30 MINUTES	0
FILLING	HOT/COLD	0
PREWASH 3 TEMPERATURE	0-95°C	0
PREWASH 3 DETERGENT	0-6 MINUTES	0
PREWASH 3 ADDITIVE 2	0-6 MINUTES	0
WASH PERIOD	0-30 MINUTES	4 MINUTES
FILLING	HOT/COLD	HOT
WASH TEMPERATURE	0-95°C	80°C
WASH DETERGENT	0-6 MINUTES	1 MINUTE. 6 SECONDS
WASH ADDITIVE 2	0-6 MINUTES	0
RUNNING WATER RINSE 1	0-9 MINUTES	2 MINUTES
ACID RINSE PERIOD	0-30 MINUTES	2 MINUTES
ACID	0-6 MINUTES	2 MINUTES
RUNNING WATER RINSE 2	0-9 MINUTES	7 MINUTES
COLD DEMI. WATER RINSE 1	0-30 MINUTES	0
COLD DEMI. WATER RINSE 2	0-30 MINUTES	0
COLD DEMI. WATER RINSE 3	0-30 MINUTES	0
COLD DEMI. WATER RINSE 4	0-3 MINUTES	0
HOT DEMI. WATER RINSE	0-30 MINUTES	2 MINUTES
HOT DEMI. TEMPERATURE	0-95°C	75°C
HOT DEMI. ADDITIVE	0-6 MINUTES	0
DRYING	0-90 MINUTES	25 MINUTES

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MATERIAL SAFETY DATA SHEET (MSDS)

VARIAN

Section 1 - Introduction

Effective Date: May 22, 1995

Manufacturer's Name: VARIAN SAMPLE PREPARATION PRODUCTS

Address: 25200 Commercentre Dr
Lake Forest, California 92630

Emergency Telephone: (800) 424-9300

Information Telephone: (949) 770-9381

Trade Name: CHEM ELUT ™ Hydromatrix

Chemical Name: FLUX CALCINED DIATOMACEOUS EARTH

Varian Reference No.: 12198001, 12198002, 12198003, 12198004, 12198005, 12198006, 12198007, 12198008, 12198009, 12198010, and 12198011

Section 2 - Hazardous Ingredients

CAS No.	Chemical Name	Percent	ACGIH TLV	OSHA PEL
68855-54-9	Diatomaceous Earth	65%	10 mg/m3	6 mg/m3
14464-46-1	Cristobalite** (crystalline silica)	35%	0.05 mg/m3	0.05 mg/m3

n/e = none established * = Sara Title III Section 313 Chemical ** = California Proposition 65 Chemical

Section 3 - Physical Description

Appearance and Odor:

Fine white powder, no odor

Melting Point: 1710 ° C

Boiling Point @ 1 atm: 2,230 ° C

Flash Point: None

Specific Gravity: 2.35

Solubility in Water: Practically Insoluble

Vapor Pressure: None

Upper Explosive Limit (UEL) in Air, % by volume: None

Lower Explosive Limit (LEL) in Air, % by volume: None

n/a = not available

Section 4 - Fire and Emergency Handling Data

Extinguishing Media:

Diatomaceous Earth is non-combustible

Personal Protective Equipment:

Use NIOSH/MSHA approved Self-Contained Breathing Apparatus (SCBA) under extremely dusty conditions.

Fire and Explosion Hazard Precautions:

When heated to extreme temperature, diatomaceous earth can crystallize or melt into glass

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MATERIAL SAFETY DATA SHEET (MSDS)

VARIAN

Section 5 - Health Hazard Data and Emergency First Aid Procedures

Routes of Entry into the Body:

Inhalation, Eye Contact, Skin

Symptoms:

Irritation and some soreness in the throat and nose. In extreme exposures some congestion may occur.

Long Term Effects:

Crystalline silica has been classified as probably carcenogenic for human by IAC. Crystalline silica is also a known cause of silicosis, a non-cancerous lung disease.

Listed as a Carcinogen or Potential Carcinogen:

NTP [N] IARC [Y] OSHA [N]

FIRST AID:

Skin Contact:

N/A

Eye Contact:

Immediately flush with water for 15 minutes

Inhalation:

Remove to fresh air. Drink water to clear throat and blow nose to remove dust.

Ingestion:

N/A

Section 6 - Reactivity Data

Stability: [ ] **Unstable** [x] **Stable**

Hazardous Polymerization: [ ] **May Occur** [x] **Will not occur**

Incompatible Materials:

Hydrofluoric Acid

Hazardous Decomposition Products:

None determined

Storage:

Store in a sealed container at room temperature.

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MATERIAL SAFETY DATA SHEET (MSDS)

VARIAN

Section 7 - Spill, Leak and Disposal Procedures

Steps To Be Taken In Case Material Is Released Or Spilled:

Vacuum dust with equipment fitted with a HEPA filter. If sweeping is necessary use a dust suppressant.

Waste Disposal Method:

Wastes are not hazardous as defined by RCRA (40 CFR Part 261.) Comply with federal, state, and local hazardous waste regulations.

Section 8 - Special Protection Information

Respiratory Protection:

Use a NIOSH-approved HEPA filter respirator is airborne levels are expected to exceed PEL or TLV limits.

Ventilation:

As needed to reduce worker exposure below PEL and TLV exposure limits.

Protective Gloves:

Not normally required.

Eye Protection:

Not normally required.

Other Protective Equipment:

Protective clothing should be worn where skin irritation or other effects may occur.

Section 9 - Product Warning Label Statement

These data are offered in good faith as typical values and not as a product specification. No warranty, either expressed or implied is hereby made. The recommended Industrial Hygiene and safety procedures are believed to be generally applicable. However, each user should review these recommendations in the specific context of the intended use and determine whether they are appropriate.

Section 10 - Other Product Warning Statements

[X] **California State Proposition 65**

This product contains a chemical known to the State of California to cause cancer, birth defects, or other reproductive harm.

[X] **SARA Title III**

This product **does not contain** a chemical listed in Section 313 of the Emergency Planning and Community Right-To-Know Act of 1986.

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MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
EQUIPMENT MONITORING RECORD
CALENDAR YEAR _____

Refrigerator/Freezer/Oven (Circle one)

Room # _____

Equipment #

Thermometer Serial #_____

Thermometer Correction Factor_____

Acceptance Range (C or F):

	JAN		FEB		MAR		APR		MAY		JUN		JUL		AUG		SEP		OCT		NOV		DEC		
Day	Temperature	Initials	Temperature	Initials	Temperature	Initials	Temperature	Initials	Temperature	Initials	Temperature	Initials	Temperature	Initials	Temperature	Initials	Temperature	Initials	Temperature	Initials	Temperature	Initials	Temperature	Initials	Day
1																									1
2																									2
3																									3
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31																									31

Recorded temperature must be observed temperature plus the thermometer correction factor.

Comments, corrective actions, & date:

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ATTACHMENT 22.16

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

INTEROFFICE COMMUNICATION

Date:

TO: (Name of your Unit Manager)
Albert Lee, QA Officer
Bob Avery, Chief, Laboratory Services Section
File, QA Officer

FROM:

SUBJECT: **Quality Assurance Corrective Action**

Please indicate the type of Quality Assurance failure/deviation:

_____ PT Study _____ Quality Assurance Data Trend _____ Other

1. **Date of failure:**
2. **Parameter(s)/Method(s) name:**
3. **Instrument:**
4. **Sample(s)/Batch(s) affected:**
   a. **Identify the sample or batch of samples affected.**
   b. **Describe if any other samples were affected by this problem and why or why not.**
5. **Detailed description of the failure/deviation:**
6. **Describe investigation of the failure/deviation.**
   a. **How was it detected, what steps did you take to resolve the problem, how was it finally resolved?**
   b. **Write a description about the QC acceptance, instrument failure, sample interference, or any other issues that affected the quality of data.**

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Attachment 22.18

MDEQ-Environmental Lab
Weekly Balance Calibration Check

A minimum of 3 weights must be checked

Date	Balance #	Analyst	1 g weight	2 g weight	5 g weight	10 g weight	20 g weight	50 g weight	100 g weight	Acceptable (yes/no)	Comments
Acceptance Criteria			+/- 0.05 g +/- 0.005 g +/- 0.0005 g	+/- 0.05 g +/- 0.005 g +/- 0.0005 g	+/- 0.05 g +/- 0.005 g +/- 0.0005 g	+/- 0.05 g +/- 0.005 g +/- 0.0005 g	+/- 0.05 g +/- 0.005 g +/- 0.0005 g	+/- 0.05 g +/- 0.010 g +/- 0.0010 g	+/- 0.05 g +/- 0.010 g +/- 0.0010 g		

COMMENTS

Acceptance criteria is dependent upon significant fiqures of balance.

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ABSOLUTE STANDARDS, INC • 44 Rossotto Drive • Hamden, CT • (800)-368-1131

ISO 9001:2000 Quality System Registered
ISO/IEC Guide 17025, ANSI Z540 Compliant
DLS ANSI-RAB Accredited

EL 2310 1-4 JA 3/2/06

Attachment 22.19

CERTIFIED WEIGHT REPORT

		Lot #	Solvent(s):
Part Number:	94206	CP114	Methanol
Lot Number:	022306		
Description:	EPA Method 625 LCS Spike		
	67 components		
Expiration Date:	022311		
Nominal Concentration (µg/mL):	Varied		

Justin Dippold 022306
Formulated By: Justin Dippold DATE

Pedro Rentas 022306
Reviewed By: Pedro L. Rentas DATE

5E-05 Balance Uncertainty

Volume(s) shown below were combined and diluted to : 500.0 0.042 Flask Uncertainty

Compound	Part Number	Lot Number	Dil. Factor	Initial Vol. (mL)	Uncertainty Pipette	Initial Conc.(ug/mL)	Final Conc.(ug/mL)	Expanded Uncertainty (+/-) (µg/mL)	CAS#	MSDS Information (Solvent Safety Info. On Attached pg.) OSHA PEL (TWA)	LD50
1. Acenaphthene	92987	072805	0.50	250.0	0.013	100.1	50.0	0.00450	00083-32-9	N/A	ipr-rat 600mg/kg
2. Acenaphthylene	92987	072805	0.50	250.0	0.013	100.1	50.1	0.00450	00208-96-8	N/A	N/A
3. Anthracene	92987	072805	0.50	250.0	0.013	100.1	50.0	0.00450	00120-12-7	0.2mg/m3 (8H)	ipr-mus 430mg/kg
4. Benzo(a)anthracene	92987	072805	0.50	250.0	0.013	100.0	50.0	0.00450	00056-55-3	N/A	N/A
5. Benzo(b)fluoranthene	92987	072805	0.50	250.0	0.013	100.3	50.1	0.00450	00205-99-2	N/A	N/A
6. Benzo(k)fluoranthene	92987	072805	0.50	250.0	0.013	100.1	50.0	0.00450	00207-08-9	N/A	orl-rat 2820mg/kg
7. Benzo(g,h,i)perylene	92987	072805	0.50	250.0	0.013	100.1	50.1	0.00450	00191-24-2	N/A	N/A
8. Benzo(a)pyrene	92987	072805	0.50	250.0	0.013	100.3	50.2	0.00453	00050-32-8	0.2mg/m3 (8H)	scu-rat 50mg/kg
9. 4-Bromophenyl phenyl ether	92987	072805	0.50	250.0	0.013	100.1	50.0	0.00450	00101-55-3	N/A	N/A
10. Benzyl butyl phthalate	92987	072805	0.50	250.0	0.013	100.3	50.1	0.00450	00085-68-7	N/A	orl-rat 2330mg/kg
11. bis(2-Chloroethoxy) methane	92987	072805	0.50	250.0	0.013	100.2	50.1	0.00450	00111-91-1	N/A	N/A
12. bis(2-Chloroethyl) ether	92987	072805	0.50	250.0	0.013	100.1	50.1	0.00450	00111-44-4	15 ppm (90mg/m3/8H)(ski	orl-rat 75mg/kg
13. bis(2-Chloroisopropyl) ether	92987	072805	0.50	250.0	0.013	100.0	50.0	0.01043	00108-60-1	N/A	orl-rat 240mg/kg
14. 2-Chloronaphthalene	92987	072805	0.50	250.0	0.013	100.1	50.0	0.00450	00091-58-7	N/A	orl-rat 2078mg/kg
15. 4-Chlorophenyl phenyl ether	92987	072805	0.50	250.0	0.013	100.2	50.1	0.00450	07005-72-3	N/A	N/A
16. Chrysene	92987	072805	0.50	250.0	0.013	100.4	50.2	0.00450	00218-01-9	0.2mg/m3	N/A
17. Di-n-butyl phthalate	92987	072805	0.50	250.0	0.013	100.4	50.2	0.00450	00084-74-2	5mg/m3/8H	orl-rat 8000mg/kg
18. Di-n-octyl phthalate	92987	072805	0.50	250.0	0.013	100.2	50.1	0.00450	00117-84-0	N/A	ipr-mus 65g/kg
19. Dibenzo(a,h)anthracene	92987	072805	0.50	250.0	0.013	100.1	50.0	0.00450	00053-70-3	0.2mg/m3	N/A
20. 1,2-Dichlorobenzene	92987	072805	0.50	250.0	0.013	100.1	50.1	0.00450	00095-50-1	50 ppm (300mg/m3) (CL)	orl-rat 500mg/kg
21. 1,3-Dichlorobenzene	92987	072805	0.50	250.0	0.013	100.1	50.1	0.00453	00541-73-1	N/A	ipr-mus 1062mg/kg
22. 1,4 Dichlorobenzene	92987	072805	0.50	250.0	0.013	100.1	50.1	0.00450	00106-46-7	75 ppm (450mg/m3/8H)	orl-rat 500mg/kg
23. 2,4-Dinitrotoluene	92987	072805	0.50	250.0	0.013	100.3	50.1	0.00456	00121-14-2	1.5mg/m3/8H (skin)	orl-rat 268mg/kg
24. 2,6-Dinitrotoluene	92987	072805	0.50	250.0	0.013	100.3	50.1	0.00450	00606-20-2	1.5mg/m3/8H (skin)	orl-rat 177mg/kg
25. Diethyl phthalate	92987	072805	0.50	250.0	0.013	100.3	50.1	0.00450	00084-66-2	5mg/m3/8H	orl-rat 8600mg/kg
26. Dimethyl phthalate	92987	072805	0.50	250.0	0.013	100.1	50.0	0.00450	00131-11-3	5mg/m3/8H	orl-rat 6800mg/kg
27. bis(2-Ethylhexyl) phthalate	92987	072805	0.50	250.0	0.013	100.1	50.0	0.00450	00117-81-7	5mg/m3/8H	orl-rat 30600mg/kg
28. Fluoranthene	92987	072805	0.50	250.0	0.013	100.2	50.1	0.00453	00206-44-0	N/A	orl-rat 2000mg/kg
29. Fluorene	92987	072805	0.50	250.0	0.013	100.1	50.0	0.00453	00086-73-7	N/A	ipr-mus 2 g/kg
30. Hexachlorobenzene	92987	072805	0.50	250.0	0.013	100.0	50.0	0.00450	00118-74-1	N/A	orl-rat 10g/kg
31. Hexachlorobutadiene	92987	072805	0.50	250.0	0.013	100.1	50.1	0.00453	00087-68-3	0.02 ppm (0.24mg/m3/8H)	orl-rat 90mg/kg
32. Hexachlorocyclopentadiene	92987	072805	0.50	250.0	0.013	100.1	50.0	0.00453	00077-47-4	0.01 ppm (0.1mg/m3/8H)	orl-rat 1300mg/kg
33. Hexachloroethane	92987	072805	0.50	250.0	0.013	100.3	50.1	0.00450	00067-72-1	1 ppm (10mg/m3/8H)(skin	orl-rat 4460mg/kg
34. Indeno(1,2,3-cd)pyrene	92987	072805	0.50	250.0	0.013	100.2	50.1	0.00450	00193-39-5	N/A	N/A
35. Isophorone	92987	072805	0.50	250.0	0.013	100.2	50.1	0.00456	00078-59-1	25 ppm	orl-rat 2330mg/kg
36. Naphthalene	92987	072805	0.50	250.0	0.013	100.3	50.1	0.00450	00091-20-3	10 ppm (50mg/m3/8H)	orl-rat 490mg/kg
37. Nitrobenzene	92987	072805	0.50	250.0	0.013	100.0	50.0	0.00450	00098-95-3	1 ppm (5mg/m3/8H)(skin)	orl-rat 780mg/kg
38. N-Nitrosodimethylamine	92987	072805	0.50	250.0	0.013	100.0	50.0	0.00453	00062-75-9	N/A	orl-rat 58mg/kg
39. N-Nitrosodi-n-propylamine	92987	072805	0.50	250.0	0.013	100.2	50.1	0.00450	00621-64-7	N/A	orl-rat 480mg/kg
40. N-Nitrosodiphenylamine	92987	072805	0.50	250.0	0.013	100.1	50.1	0.00453	00086-30-6	N/A	orl-rat 2140mg/kg
41. Phenanthrene	92987	072805	0.50	250.0	0.013	100.2	50.1	0.00450	00085-01-8	0.2mg/m3/8H	orl-mus 700mg/kg
42. Pyrene	92987	072805	0.50	250.0	0.013	100.2	50.1	0.00453	00129-00-0	0.2mg/m3/8H	orl-rat 2700mg/kg
43. 1,2,4-Trichlorobenzene	92987	072805	0.50	250.0	0.013	100.1	50.0	0.00450	00120-82-1	5 ppm (CL) (40mg/m3)	orl-rat 756mg/kg
44. Aniline	94208	022306	0.05	25.0	0.011	1001.1	50.1	0.00417	00062-53-3	5 ppm (8H)	orl-rat 250mg/kg
45. Azobenzene	94208	022306	0.05	25.0	0.011	1002.4	50.1	0.00428	00103-33-3	N/A	orl-rat 1000mg/kg
46. Carbazole	94208	022306	0.05	25.0	0.011	1003.0	50.1	0.00416	00086-74-8	N/A	ipr-mus 200mg/kg
47. Dibenzofuran	94208	022306	0.05	25.0	0.011	1002.5	50.1	0.00417	00132-64-9	N/A	N/A
48. 2-Methylnaphthalene	94208	022306	0.05	25.0	0.011	1002.2	50.1	0.00428	00091-57-6	N/A	orl-rat 1630mg/kg
49. 2-Nitroaniline	94208	022306	0.05	25.0	0.011	1001.0	50.0	0.00420	00088-74-4	N/A	orl-rat 1600mg/kg
50. 3-Nitroaniline	94208	022306	0.05	25.0	0.011	1002.6	50.1	0.00420	00099-09-2	N/A	orl-rat 535mg/kg
51. 4-Nitroaniline	94208	022306	0.05	25.0	0.011	1001.4	50.1	0.00420	00100-01-6	1 ppm (6mg/m3/8H)(skin)	orl-rat 750mg/kg
52. Pyridine	94208	022306	0.05	25.0	0.011	1003.1	50.2	0.00414	00110-86-1	5 ppm (15mg/m3/8H)	orl-rat 891mg/kg
53. 4-Chloro-3-methylphenol	94196	021306	0.05	25.0	0.011	2002.3	100.1	0.00435	00059-50-7	N/A	orl-rat 1830mg/kg
54. 2-Chlorophenol	94196	021306	0.05	25.0	0.011	2000.6	100.0	0.00435	00095-57-8	N/A	orl-rat 670mg/kg
55. 2,4-Dichlorophenol	94196	021306	0.05	25.0	0.011	2001.8	100.1	0.00435	00120-83-2	N/A	orl-rat 580mg/kg
56. 2,4-Dimethylphenol	94196	021306	0.05	25.0	0.011	2001.9	100.1	0.00443	00105-67-9	N/A	orl-rat 3200mg/kg
57. 2,4-Dinitrophenol	94196	021306	0.05	25.0	0.011	2000.6	100.0	0.00439	00051-28-5	N/A	orl-rat 30mg/kg
58. 4,6-Dinitro-2-methylphenol	94196	021306	0.05	25.0	0.011	2001.1	100.0	0.00439	00534-52-1	0.2mg/m3/8H (skin)	orl-rat 10mg/kg
59. 2-Nitrophenol	94196	021306	0.05	25.0	0.011	2003.4	100.2	0.00439	00088-75-5	N/A	orl-rat 334mg/kg
60. 4-Nitrophenol	94196	021306	0.05	25.0	0.011	2002.4	100.1	0.00439	00100-02-7	N/A	orl-rat 250mg/kg
61. Pentachlorophenol	94196	021306	0.05	25.0	0.011	2002.4	100.1	0.00439	00087-86-5	0.5mg/m3/8H (skin)	orl-rat 27mg/kg
62. Phenol	94196	021306	0.05	25.0	0.011	2001.3	100.1	0.00435	00108-95-2	5 ppm (19mg/m3/8H)(skin	orl-rat 317mg/kg
63. 2,4,6-Trichlorophenol	94196	021306	0.05	25.0	0.011	2002.0	100.1	0.00435	00088-06-2	N/A	orl-rat 820mg/kg
64. Benzyl alcohol	94207	022306	0.05	25.0	0.011	2001.2	100.0	0.00408	00100-51-6	N/A	orl-rat 1230mg/kg
65. o-Cresol (2-Methylphenol)	94207	022306	0.05	25.0	0.011	2001.6	100.1	0.00408	00095-48-7	5 ppm (22mg/m3/8H)(skin	orl-rat 121mg/kg
66. p-Cresol (4-Methylphenol)	94207	022306	0.05	25.0	0.011	2000.7	100.0	0.00408	00106-44-5	5 ppm (22mg/m3/8H)(skin	orl-rat 207mg/kg
67. 2,4,5-Trichlorophenol	94207	022306	0.05	25.0	0.011	2002.1	100.1	0.00408	00095-95-4	N/A	orl-rat 820mg/kg

Michigan Department of Environmental Quality

Laboratory Services Standard Operating Procedure Document

EFFECTIVE DATE: 05/2006 SOP# 402 REVISION # 1

SILICA GEL CLEANUP OF PCBs (AS AROCLORS) AND ORGANOCHLORINE PESTICIDES

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1.0 Applicable Analytical Methods

1.1 Silica Gel Cleanup, U.S. Environmental Protection Agency, Method 3630C, SW846, Revision 3, December 1996.

1.2 Quality Control, U.S. Environmental Protection Agency, Chapter 1, SW846, Revision 1, July 1992.

1.3 Organic Extraction and Sample Preparation, U.S. Environmental Protection Agency, Method 3500B, SW846, Revision 2, December 1996.

1.4 Cleanup, U.S. Environmental Protection Agency, Method 3600C, SW846, Revision 3, December 1996.

2.0 Matrix or Matrices

2.1 Aqueous.

2.2 Soil.

2.3 Sediment.

2.4 Sludge.

2.5 Clay.

2.6 Oil.

2.7 Solid or liquid waste.

3.0 Method Detection Limits (MDLs)

3.1 All MDLs are performed according to the Code of Federal Regulations 40 CFR, Part 136, Appendix B. See internal procedure SOP 103.

3.2 MDLs are extracted according to SOP 401, SOP 404, or SOP 405 and cleaned by following SOP 400 and/or SOP 403 prior to silica gel cleanup. The extract must be in hexane.

4.0 Scope and Application

4.1 This method provides for the column or cartridge cleanup of organochlorine pesticides and PCBs (as aroclors) from other interfering compounds.

4.2 This method separates most single component organochlorine pesticides from the aroclors. See Attachment 22.9 for compound distribution in each fraction.

4.3 Silica gel is an adsorbent with weakly acidic properties and can separate compounds of different chemical polarity.

4.4 Silica gel columns, with 5 grams of silica gel, are used when the pesticide (2nd fraction) needs to be collected and silica gel cartridges, with 1 gram of silica gel, are used when only the PCB fraction (1st fraction) needs to be collected.

4.5 All oil samples and sediment low level PCBs are cleaned by silica gel.

5.0 Method Summary

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5.1 Water samples are extracted by following SOP 405 and cleaned up as in SOP 400 and SOP 403. Solid samples are extracted by following SOP 401 and cleaned by following SOP 400. Oil samples are prepared by following SOP 404 and cleaned by following SOP 400. The sample extracts are then solvent exchanged to hexane.

5.2 Activated silica gel is weighed and packed into a chromatography column with a small layer of sodium sulfate on the top. The column is rinsed with appropriate solvents. If SPE cartridges are used, they are place on the Supelco SPE vacuum manifold and rinsed with appropriate solvents.

5.3 Sample extracts are introduced onto the top of the prepared column along with azulene. Or samples are introduced onto SPE cartridges without azulene as only the first fraction is collected. Azulene is used to help visually identify the end of the fraction I. Collection tubes are replaced with new ones to collect fraction II. Both fractions are concentrated to 1 mL in hexane and analyzed by gas chromatography with electron capture detection (GC-ECD)(SOP 502).

6.0 Definitions

6.1 Method Preparation Blank (MPB) - An aliquot of a blank matrix that is treated exactly as a sample, including exposure to all glassware, equipment, solvents, reagents, and surrogates that are used with samples.

6.2 Laboratory Control Sample (LCS) - An aliquot of blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is treated exactly as a sample, including extraction, cleanup, and analysis.

6.3 Matrix Spike (MS) - An aliquot of a sample to which known quantities of the target analytes are added in the laboratory. The MS is treated exactly as a sample, including extraction, cleanup, and analysis. The background concentrations of the analytes in the sample must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

6.4 Matrix Spike Duplicate (MSD) - A second aliquot of sample to which known quantities of the target analytes are added in the laboratory. The MSD is treated exactly as a sample, including extraction, cleanup, and analysis. The background concentrations of the analytes in the sample must be determined in a separate aliquot and the measured values in the MSD corrected for background concentrations. Analyses of the MS and MSD indicate precision associated with laboratory procedures.

7.0 Interferences

7.1 Interferences from contaminants can occur from solvents, glassware, and other sample processing equipment. Pesticide quality solvents (or better) and solvent rinsing of all glassware should help minimize contaminants.

7.2 The use of plastic must be minimized to prevent phthalate contamination.

7.3 Soap residue must be thoroughly rinsed from all glassware to avoid degradation of some pesticide compounds.

8.0 Safety

8.1 Safety glasses are required in all designated laboratory areas.

8.2 Be familiar with the Laboratory Chemical Hygiene Plan.

8.3 Be familiar with the Laboratory Safety Policy (SOP 100).

8.4 Hexane, toluene, azulene, silica gel, and sodium sulfate material safety data sheets (MSDSs) are attached.

8.5 Nitrile or other solvent resistant gloves must be worn when handling solvents.

9.0 Equipment and Supplies

9.1 Chromatographic columns – 9 mm x 200 mm, with a 50 mL reservoir.

9.2 Vials - 2 mL crimp cap with caps.

9.2.1 Place 1 mL of hexane into each vial from calibrated bottle-top dispenser
9.2.2 Mark the meniscus on each vial with a lab marker.
9.2.3 Dispose of the hexane. Let vial air dry.

9.3 Turbovap tubes from Caliper 250 mL and 50 mL capacity and suitable racks.

9.4 Turbovap concentration workstation from Caliper - with temperature control up to 95°C and nitrogen pressure from zero to 20 psi (settings: temperature = 35°C and pressure = 15 psi).

9.5 Glass wool - Pyrex brand or equivalent.

9.6 Pasteur pipets.

9.7 Muffle furnace - capable of attaining 400°C.

9.8 Oven - capable of maintaining 130°C.

9.9 Aluminum weigh boats.

9.10 Balance - capable of weighing to 0.01g.

9.11 Bottle top dispensers - 1 to 10 mL.

9.12 Graduated cylinders - various sizes.

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9.13 Syringes - various sizes.

9.14 Volumetric flasks - various sizes.

9.15 Amber bottles - 125 mL, with teflon lined caps.

9.16 Beakers – 100 mL, pyrex brand.

9.17 Screw cap test tubes – 13 X 100

9.17.1 Place 2 mL of hexane into each tube from calibrated bottle-top dispenser
9.17.2 Mark the meniscus with a lab marker.
9.17.3 Dispose of the hexane. Let tube air dry.

9.18 Glass test tubes – 16 X 100mm

9.19 Supelco SPE Vacuum Manifold – 8 port or larger or equivalent

9.20 Solid phase extraction cartridges – Strata SI-1, part #8B-SO12-JCH with silica 55um, 70A from Phenomenex or equivalent

10.0 Reagents and Standards

10.1 All reagents, solvents, and standards must be traceable to the stock inventory tracking log.

10.2 All reagents, solvents, and standards must be labeled with the date received, the date opened, the expiration date, the tracking number, and the receiver's initials.

10.3 All prepared reagents and standards must be labeled with the date prepared, the expiration date, the preparer's initials, the tracking number, the diluent, and the description.

10.4 All standard logbooks must be completely filled out.

10.5 Hexane (95% n-Hexane) - pesticide grade, demonstrated to be free of target analytes.

10.6 Toluene - Nanograde, demonstrated to be free of target analytes.

10.7 Azulene (cyclopentacycloheptene) - Sigma-Aldrich, part # 4273 (99%).

10.7.1 Weigh 500 mg of azulene into a 100 mL volumetric flask.
10.7.2 Dilute to the mark with hexane (Section 10.5). Mix.
10.7.3 Transfer to an amber bottle with a teflon lined cap. Label.
10.7.4 Fill out the semivolatile standard logbook. See Attachment 22.2.
10.7.5 Store in a refrigerator. There is no expiration date for azulene.

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10.8 Silica gel - 60, 70-230 mesh, Mallinckrodt part #V153, special for column chromatography or equivalent.

10.8.1 Place silica gel in beaker (Section 9.16).
10.8.2 Place the beaker in a muffle furnace at 400°C for 12 hours.
10.8.3 Cool in the muffle furnace. Transfer to a glass jar.
10.8.4 Store in an oven at 130°C.

10.9 Sodium sulfate - ACS grade, granular, anhydrous.

10.9.1 Transfer to a glass beaker.
10.9.2 Place in an oven at 130°C for at least 12 hours.

10.10 Stock Standards (SS) – All standards must be certified reference materials. Manufacturer's certificates of analysis are retained by the laboratory and kept as part of the overall QA/QC requirements. All SSs must be given a unique lab tracking number and logged into the SS (EL) tracking logbook. (Attachment 22.1) Store SSs in amber sealed ampules. Protect the standards from light. All standards are received in hexane unless otherwise noted.

10.10.1 SSs – SSs are purchased as mixes in sealed ampules and stored in a freezer at a temperature lower than 0°C.

10.10.1.1 Toxaphene (1000 ug/mL) in hexane– Absolute Stds, part #20021 or equivalent.

10.10.1.2 PCB mix Aroclors 1016 and 1260 (1000 ug/mL) in hexane – Absolute Standards, part #20064 or equivalent.

10.10.1.3 Custom pesticide standard (1000 ug/mL) in hexane:toluene - Accustandard Inc., part #S-7179-R1 or equivalent.

Aldrin	Endosulfan Sulfate
alpha-BHC	Endrin
beta-BHC	Endrin Aldehyde
delta-BHC	Endrin Ketone
gamma-BHC (Lindane)	Heptaclor
alpha-Chlordane	Heptaclor Epoxide
gamma-Chlordane	Methoxyclor
4,4'-DDD	Mirex
4,4'-DDE	Hexabromobenzene
4,4'-DDT	PBB (BP-6)
Dieldrin	TCMX (surrogate)
Endosulfan I	DCB (surrogate)
Endosulfan II	

10.10.1.4 Custom VOC Standard (1000 ug/mL) in Acetone - Accustandard Inc., part #S-8258 or equivalent.

Pentachlorobenzene	Hexachlorobenzene
Pentachloronitrobenzene	Hexachlorobutadiene
1,2,3,4-Tetrachlorobenzene	Hexachlorocyclopentadiene
1,2,4,5-Tetrachlorobenzene	

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10.10.1.5 Surrogate mix (200ug/mL) in acetone – Absolute Standards, part #20023 or equivalent – 2,4,5,6-Tetrachloro-m-xylene and Decachlorobiphenyl

10.11 Intermediate and working standards (WS) – All prepared standards must be given a unique laboratory tracking number and logged into the semivolatile standards logbook (Attachment 22.2). Store prepared standards in labeled amber bottles in a freezer. Prepared standards are good for up to six months.

10.11.1 Intermediate toxaphene standard (4000 ug/L) – Place hexane into a 25 mL volumetric flask. Inject 100 uL of toxaphene SS (Section 10.10.1.1) directly into the hexane in the flask. Bring to volume with hexane. Cap. Mix.

10.11.2 Toxaphene WS (500 ug/L) – Place hexane into a 50 mL volumetric flask. Pipet or inject 6.25 mL of intermediate toxaphene standard (Section 10.11.1) directly into the hexane in the flask. Bring to volume with hexane. Cap. Mix.

10.11.3 Intermediate aroclor standard (2000 ug/L) – Place hexane into a 500 mL volumetric flask. Inject 1mL of aroclor 1016 and 1260 mixture SS (Section 10.10.1.2) directly into the hexane in the flask. Bring to volume with hexane. Cap. Mix.

10.11.4 Aroclor WS (500 ug/L) – Place hexane into a 200 mL volumetric flask. Pipet or inject 50 mL of intermediate aroclor standard (Section 10.11.3) directly into the hexane in the flask. Bring to volume with hexane. Cap. Mix.

10.11.5 Pesticide WS (250 ug/L) – Place hexane into a 500 mL volumetric flask. Inject 125 uL of custom pesticide SS (Section 10.10.1.3) and 125 uL of custom VOC pesticide SS (Section 10.10.1.4) directly into the hexane in the flask. Bring to volume with hexane. Cap. Mix.

10.11.6 Aroclor cartridge check standard (100ug/L) – Place hexane into a 200 mL volumetric flask. Inject 10mL of intermediate aroclor standard (Section 10.11.3) directly into the hexane in the flask. Bring to volume with hexane. Cap. Mix.

10.11.7 Pesticide cartridge check standard (125ug/L) – Place hexane into a 200 mL volumetric flask. Inject 100 mL of pesticide WS (Section 10.11.5) directly into the hexane in the flask. Bring to volume with hexane. Cap. Mix.

10.11.8 Surrogate mix (200ug/L) – Place hexane into 500mL volumetric flask. Inject 0.5mL of surrogate mix (Section 10.10.1.5) directly into hexane in flask. Bring to volume with hexane. Cap. Mix.

11.0 Sample Collection, Preservation, Shipment and Storage

11.1 Aqueous samples – see SOP 405.

11.2 Solid samples – see SOP 401.

11.3 Waste samples – see SOP 404.

11.4 Holding times – Sample extracts may be analyzed for up to 40 days from the extraction date.

11.5 Storage – Sample extracts must be stored in a freezer and protected from light in crimp cap vials until analysis.

12.0 Quality Control (QC)

12.1 All associated QC samples must be processed through this cleaning method along with the sample extracts.

NOTE: *The MS/MSDs do not need to go through this cleaning procedure if the sample they are associated with does not need this cleaning.*

12.2 Extracted sample batches.

12.2.1 Extracted sample batches consist of a maximum of 20 samples, 1 MPB, 1 MS, 1 MSD, and 1 LCS (if a MS/MSD is not available, a laboratory control sample duplicate [LCSD] must be analyzed).

12.2.2 MSs and MSDs are actual samples that are selected at random from within the batch of 20.

12.2.3 All 20 samples and the QC samples are spiked with known surrogate concentrations. The recoveries for these compounds are used as indicators of possible problems that may occur during extraction or during cleanup of individual samples.

12.2.4 The MS, MSD, LCS, and LCSD are spiked at a known concentration with target analyte compounds. The replicate MSs or LCSs are used to evaluate the entire batch for precision and accuracy.

12.3 Initial demonstration of capability (IDOC) - Each analyst must demonstrate initial proficiency with this method by generating data of acceptable accuracy and precision for the target analytes. This is accomplished by extraction and cleanup of four aliquots of a reference sample.

12.3.1 Five aliquots of reagent water or sand are extracted using SOP 401 or SOP 405. One is a MPB, and the other four are spiked with a mid-range concentration of all the pesticide analytes of interest (10.11.5).

12.3.2 Sample extracts are taken through all the normal sample cleaning methods.

12.3.3 Extracts are then analyzed by GC-ECD. See SOP 502 for further details.

13.0 Calibration and Standardization

13.1 Each new lot of silica gel is tested to determine the separation point between fraction I and fraction II. The procedure is as follows:

13.1.1 Prepare ten columns with the new silica gel (Section 14.1.6).

13.1.2 Premark three columns at 1 cm above the glass wool.

13.1.3 Premark four columns at 1.5 cm above the glass wool.

13.1.4 Premark three columns at 2 cm above the glass wool.

13.1.5 Place ten screw cap tubes in a rack. Add 2 mL of hexane (Section 10.5) to each tube. Label one tube as blank. Label three tubes as toxaphene. Label three tubes as aroclors. Label three tubes as pesticides.

NOTE: *The blank is fractionated at the 1.5 cm mark. One of each of the other solutions is fractionated at the 1 cm mark, the 1.5 cm, and the 2 cm mark.*

13.1.6 Inject 200 uL of toxaphene standard (Section 10.11.2) into the test tubes labeled toxaphene.
13.1.7 Inject 200 uL of aroclor standard (Section 10.11.4) into the test tubes labeled aroclors.
13.1.8 Inject 200 uL of pesticide standard (Section 10.11.5) into the test tubes labeled pesticides.
13.1.9 Add 200 uL of azulene (Section 10.7.3) to all 10 test tubes.
13.1.10 Add 500ul of surrogate(Section10.11.8) to the test tube labeled blank.
13.1.11 Proceed to Section 14.3.6.
13.1.12 See Attachment 22.9 for desired fractionation. If fractionation is not optimum, repeat above fractionation at other markings. Repeat until optimum fractionation is achieved.
13.1.13 Total recovery is determined by adding the results of both fractions together. Total recovery must be >85% for every compound. The lot is rejected if total recovery is not >85% for every compound.
13.1.14 Record results of these lot tests in the silica gel run logbook. The optimum cm column markings for the lot must be indicated.
13.1.15 The predetermined mark for this lot of silica gel is good until replaced with a new lot.

13.2 Each new lot number of SPE cartridges is tested to determine sufficient recovery of the PCBs and 1st fraction pesticides. Because azulene moves through too rapidly in the cartridge, the cartridges are not used for separating the 2nd fraction pesticides.

13.2.1 Five cartridges are placed on the SPE vacuum manifold.
13.2.2 Add 2 mL of a mix of standards (5 mL of Section 10.11.6 and 5 mL of Section 10.11.7) to each of four cartridges and 500uL of surrogate (Section 10.11.8) in 2 ml of hexane to the fifth cartridge for the MPB. Use procedure specified for cartridges (Section 14.2.3).
13.2.3 Recoveries of analytes must be at least 85% of spiked amount and free of interferences for cartridge lot to be accepted.

14.0 Procedure

14.1 Procedure for Silica Gel Columns.

14.1.1 This procedure must be performed in an area where the temperature and humidity can be controlled.
14.1.2 The optimum humidity is 25-35%.
14.1.3 The optimum temperature is 20-26°C (68–79°F).
14.1.4 Cold drafts must be avoided as they cause channeling within the column.
14.1.5 Sample preparation.

14.1.5.1 Extracts must be in hexane.
14.1.5.2 Extracts are transferred to 13X100 screw top tubes (section 9.17.3) and brought up to 2 mL with hexane.

NOTE: *If the extract is less than 1 mL, the exact volume must be measured using a syringe prior to transfer. Record the initial extract volume in the silica gel logbook (Attachment 22.3). Alternately, the extract level may be marked on the vial and the vial reused to collect the new extract.*

14.1.5.3 Allow extracts to reach room temperature.
14.1.5.4 Add 200 uL of azulene (Section 10.7.3) to every extract requiring cleanup.

14.1.6 Column Preparation.

14.1.6.1 Place some glass wool in the end of a chromatographic column.
14.1.6.2 Measure and mark the column at the predetermined level for the current lot number above the glass wool (Section13.1).
14.1.6.3 Weigh 5 g (± 0.01 g) of hot silica gel (Section 10.8.4) into an aluminum weigh boat.
14.1.6.4 Transfer the weighed silica gel to the column. Tap vigorously against wooden stand to pack the silica gel.
14.1.6.5 Add approximately 1 cm of hot sodium sulfate (Section 10.9.2) to the top of the column.

NOTE: *Pack three to four columns to allow the silica gel to cool slightly. Do not pack more than four columns or channeling could result.*

14.1.6.6 Rinse the column with 10 mL of hexane (Section 10.5). The column is ready when it appears clear with no sign of channeling. Let elute until just above level of sodium sulfate.

NOTE: *Do not let the columns go dry.*

14.1.6.7 Immediately proceed to Section 14.1.7.

14.1.7 Procedure.

14.1.7.1 Pour extract (Section 14.1.5.4) onto the column.
14.1.7.2 Rinse extract vial with 2 mL of hexane. Add this to the column just prior to the column going dry.
14.1.7.3 Repeat Section 14.1.7.2.
14.1.7.4 Prior to exposure of the column to air, discard eluate. Add 35 mL of hexane. Place a 250 mL turbovap tube under the column to collect fraction I.
14.1.7.5 Collect fraction I until the bottom of the azulene reaches the predetermined mark (Section 14.1.6.2).

NOTE: *Add additional hexane if necessary.*

14.1.7.6 Change turbovap tubes to collect fraction II. If any hexane remains in the reservoir, let it drain into fraction II or pipet it off the column and dispose.
14.1.7.7 Prior to exposure of the column to air, add 45 mL of toluene (Section 10.6).

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NOTE: *Toluene causes the column to become clear, allowing the observance of the migration of the solvent through the column. Note any channeling.*

14.1.7.8 The column may be allowed to go to dryness at this point.
14.1.7.9 Concentrate each fraction on the turbovap workstation to less than 1 mL.
14.1.7.10 Transfer each fraction to 1 mL premarked crimp cap vials (Section 9.2) using pasteur pipets. Dilute to 1 mL with hexane. Cap.
14.1.7.11 Label each vial with sample number and fraction I or II.
14.1.7.12 Copy the silica gel logbook page onto blue colored paper.
14.1.7.13 Store extracts in the pesticide/PCB sample storage freezer. Staple the blue logbook copy to the batch sheets. Give this and the extracts to the analyst.

14.2 Procedure for Silica Gel Cartridges

14.2.1 Sample preparation will be the same as in Section 14.1.5. (Skip step 14.1.5.4)
14.2.2 Cartridge preparation.

14.2.2.1 Attach cartridges to flow control valves on top of SPE vacuum manifold.
14.2.2.2 Put test tubes under each cartridge in manifold to collect waste.
14.2.2.3 Set vacuum to ten inches of Hg and add 6 mL of hexane to each cartridge.
14.2.2.4 Open valve to allow a few drops to flow out, then close and let sit for at least 5 minutes.

14.2.3 Procedure

14.2.3.1 Open valves and let hexane elute at about 2 mL per minute until level is about 1 mm above the silica gel in the cartridge.
14.2.3.2 Transfer 2 mL of sample extract (Section 14.1.5.3) to cartridge. Let sample elute at about 2 mL per minute until just above silica gel. Do not let it go dry. Close control valves.
14.2.3.3 Rinse sample vial with about 0.5 mL hexane and add to cartridge.
14.2.3.4 Turn off vacuum, remove waste tubes and replace with labeled 16x100 test tubes.
14.2.3.5 Turn on vacuum and open valves to let solvent through, but do not let them go dry.
14.2.3.6 Close valves, add 5 mL hexane to cartridges and let stand for 1 minute.
14.2.3.7 Open valves and let elute until hexane just reaches top of silica gel in cartridge.
14.2.3.8 Turn off vacuum, remove collection tubes, and pour each sample into a labeled 50 mL capacity turbovap tube.
14.2.3.9 Rinse each collection tube with 5 mL hexane and add to turbovap tubes.
14.2.3.10 Concentrate on the turbovap workstation to less than 1 mL.
14.2.3.11 Transfer each fraction to 1 mL pre-marked crimp cap vials (Section 9.2) using Pasteur pipets. (Section 9.6) 'Dilute to 1mL with hexane. Cap vial.
14.2.3.12 Label each vial with sample number.
14.2.3.13 Copy the silica gel logbook page onto blue colored paper.
14.2.3.14 Store extracts in the pesticide/PCB sample storage freezer. Staple the blue logbook copy to the batch sheets. Give this and the extracts to the analyst.

14.3 Glassware cleaning

14.3.1 Use house air line to blow used silica gel from columns.
14.3.2 Put columns and turbovap tubes in dishwasher.
14.3.3 Run dishwasher as directed (Attachment 22.10).
14.3.4 Rinse all glassware with hexane prior to use.

15.0 Calculations

15.1 Divide the recovered concentration by the spiked amount and multiply by 100 to find the % recovery of each compound.
15.2 IDOC – The average and SD are determined and the % RSD is calculated by dividing the SD by the average and multiplying by 100.

16.0 Method Performance

16.1 Each MDL study must be labeled with the analyst's name and the date.

16.2 Each IDOC study must be labeled with the analyst's name and the date. A copy must be placed into the analyst's training file.

17.0 Pollution Prevention

17.1 This method uses small quantities of organic solvents. The waste that is produced is collected for disposal. (See SOP 111)

17.2 For information concerning pollution prevention that may be applicable to laboratory operations, consult Less is Better: Laboratory Chemical Management for Waste Reduction available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, DC 20036.

18.0 Data Assessment

18.1 See SOP 502.

19.0 Corrective Actions

19.1 QA/QC failure – If the QA/QC in SOP 502 is not met and it is determined to be attributed to the extraction or clean up, samples may be repeated through this cleaning process.

20.0 Waste Management

20.1 The Laboratory Safety Officer arranges for proper waste disposal.

UNCONTROLLED DOCUMENT

21.0 References

21.1 United States Code of Federal Regulations, Title 40, U.S. Government Printing Office, Washington, DC, Appendix A and B.

21.2 Test Methods for Evaluating Solid Waste, U.S. Environmental Protection Agency, Document SW-846, Third Edition 1986, Final Update III, December 1996.

22.0 Attachments

22.1 Stock standard logbook page.

22.2 Semivolatile standard logbook page.

22.3 Silica gel runlog page.

22.4 Hexane MSDS.

22.5 Azulene MSDS.

22.6 Toluene MSDS.

22.7 Silica gel MSDS.

22.8 Sodium sulfate MSDS.

22.9 Distribution of organochlorine pesticides and PCBs (as aroclors) in silica gel column fractions.

22.10 Dishwasher program.

UNCONTROLLED DOCUMENT

Signature Page

APPROVED BY _____ DATE 5/4/06
CHIEF, LABORATORY SERVICES SECTION

APPROVED BY _____ DATE 5/4/06
COMPLIANCE OFFICER

APPROVED BY _____ DATE 3/28/06
QA OFFICER

APPROVED BY _____ DATE 3-22-06
UNIT MANAGER

APPROVED BY _____ DATE 3/23/06
ANALYST

MDEQ-ENVIRONMENTAL LAB
STOCK TRACKING LOG

Environmental Lab Tracking Number	QTY (each)	Filley Tracking Number	Date Received (at lab)	Receiver Initials	Chemical,Analyte, Reagent or Solvent	Vendor and Catalog #	PO #	Manufacturer and Lot Number	Expiration Date
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
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EL-									
EL-									

ATTACHMENT 22.1

FILE:ST-STD
12/30/98

MDEQ-ENVIRONMENTAL LAB
SEMI-VOLATILE ORGANIC STANDARDS

SVOC Tracking Number	Date Prepared	EL or SVOC Tracking Number	Analyte or Mix	Preparers Initials	Solvent	Solvent Tracking Number	Initial Concentration*	Initial Volume/Wt Standard*	Final Volume*	Final Concentration*	Expiration Date
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
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SE-											
SE-											
SE-											
SE-											
SE-											
SE-											

ATTACHMENT 22.2

FILE:SV-STD
12/30/98

Scan:_____
Method: 3620
Date:_____
Analyst:_____
Batch #_____

MDEQ-Environmental Lab
Silica Gel Cleanup Logbook-Organics

Humidity _____
Temperature_____

ATTACHMENT 22.3

	Silica Gel #	Hexane #			Toluene #		Azulene #
#	Sample #	Fraction #	Extraction Batch #	Volume of Toluene (mL)	Volume of Extract Cleaned (uL)	Final Volume (uL)	Comments
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							

COMMENTS

ATTACHMENT 22.4

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

HEXANES

MSDS Number: H2379 --- *Effective Date: 04/15/99*

1. Product Identification

Synonyms: Normal Hexane; Hexyl Hydride
CAS No.: 110-54-3 (n-hexane)
Molecular Weight: 86.18
Chemical Formula: CH3(CH2)4CH3 n-hexane
Product Codes: 9277, 9306, 9309, N169

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Hexane	110-54-3	> 95%	Yes
Methylcyclopentane	96-37-7	1 - 2%	Yes
2-Methylpentane	107-83-5	1 - 2%	No
3-Methylpentane	96-14-0	1 - 2%	No
Pentane	109-66-0	0 - 2%	No

3. Hazards Identification

Emergency Overview

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.

ATTACHMENT 22.4

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Life)
Flammability Rating: 4 - Extreme (Flammable)
Reactivity Rating: 2 - Moderate
Contact Rating: 2 - Moderate
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

The health hazards addressed are for the major component: n-hexane.

Inhalation;
Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Greater exposure may cause muscle weakness, numbness of the extremities, unconsciousness and death.
Ingestion:
May produce abdominal pain, nausea. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms expected to parallel inhalation.
Skin Contact:
May cause redness, irritation, with dryness, cracking.
Eye Contact:
Vapors may cause irritation. Splashes may cause redness and pain.
Chronic Exposure:
Repeated or prolonged skin contact may defat the skin and produce irritation and dermatitis. Chronic inhalation may cause peripheral nerve disorders and central nervous system effects.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance. May affect the developing fetus.

---

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:
Aspiration hazard. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Get medical attention

immediately.
Skin Contact:
Remove any contaminated clothing. Wipe off excess from skin. Wash skin with soap and water for at least 15 minutes. Get medical attention if irritation develops or persists.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

Note to Physician:
BEI=2,5-hexadione in urine, sample at end of shift at workweeks end, 5 mg/g creatine. Also, measure n-hexane in expired air. Analgesics may be necessary for pain management, there is no specific antidote. Monitor arterial blood gases in cases of severe aspiration.

---

5. Fire Fighting Measures

Fire:
Flash point: -21C (-6F) CC
Autoignition temperature: 224C (435F)
Flammable limits in air % by volume:
lel: 1.1; uel: 7.5
Extremely Flammable Liquid and Vapor! Vapor may cause flash fire. Dangerous fire hazard when exposed to heat or flame.
Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Contact with oxidizing materials may cause extremely violent combustion.Explodes when mixed @ 28C with dinitrogen tetraoxide. Sensitive to static discharge.
Fire Extinguishing Media:
Dry chemical, foam or carbon dioxide. Water may be ineffective.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Water spray may be used to keep fire exposed containers cool. Vapors can flow along surfaces to distant ignition source and flash back. Vapor explosion hazard exists indoors, outdoors, or in sewers.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel

attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB(R) solvent adsorbent is recommended for spills of this product.

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7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from direct sunlight and any area where the fire hazard may be acute. Store in tightly closed containers (preferably under nitrogen atmosphere). Outside or detached storage is preferred. Inside storage should be in a standard flammable liquids storage room or cabinet. Separate from oxidizing materials. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
N-Hexane [110-54-3]:
-OSHA Permissible Exposure Limit (PEL): 500 ppm (TWA)
-ACGIH Threshold Limit Value (TLV): 50 ppm (TWA), Skin
other isomers of hexane
-ACGIH Threshold Limit Value (TLV): 500 ppm (TWA),1000ppm (STEL)
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. This substance has poor warning properties.
Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.
Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

The following properties are for n-hexane, the major component.
Appearance:
Clear, colorless liquid.
Odor:
Gasoline-like odor.
Solubility:
Insoluble in water.
Specific Gravity:
0.66
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100
Boiling Point:
ca. 68C (ca. 154F)
Melting Point:
ca. -95C (ca. -139F)
Vapor Density (Air=1):
3.0
Vapor Pressure (mm Hg):
150 @ 25C (77F)
Evaporation Rate (BuAc=1):
9

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Heat will contribute to instability.
Hazardous Decomposition Products:
May produce acrid smoke and irritating fumes when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Strong oxidizers.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

11. Toxicological Information

N-Hexane: Oral rat LD50: 28710 mg/kg. Irritation eye rabbit: 10 mg mild. Investigated as a tumorigen, mutagen and reproductive effector.

ATTACHMENT 22.4

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Hexane (110-54-3)	No	No	None
Methylcyclopentane (96-37-7)	No	No	None
2-Methylpentane (107-83-5)	No	No	None
3-Methylpentane (96-14-0)	No	No	None
Pentane (109-66-0)	No	No	None

12. Ecological Information

Environmental Fate:
When released into the soil, this material may biodegrade to a moderate extent. When released into the soil, this material is not expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material may biodegrade to a moderate extent. When released to water, this material is expected to quickly evaporate. When released into the water, this material is expected to have a half-life between 1 and 10 days. This material has an estimated bioconcentration factor (BCF) of less than 100. This material has a log octanol-water partition coefficient of greater than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material is expected to be readily degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material is expected to have a half-life between 1 and 10 days.
Environmental Toxicity:
No information found.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: HEXANES
Hazard Class: 3
UN/NA: UN1208
Packing Group: II
Information reported for product/size: 215L

ATTACHMENT 22.4

International (Water, I.M.O.)

Proper Shipping Name: HEXANES
Hazard Class: 3.1
UN/NA: UN1208
Packing Group: II
Information reported for product/size: 215L

---

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Hexane (110-54-3)	Yes	Yes	Yes	Yes
Methylcyclopentane (96-37-7)	Yes	Yes	No	Yes
2-Methylpentane (107-83-5)	Yes	Yes	Yes	Yes
3-Methylpentane (96-14-0)	Yes	Yes	Yes	Yes
Pentane (109-66-0)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Hexane (110-54-3)	Yes	Yes	No	Yes
Methylcyclopentane (96-37-7)	Yes	Yes	No	Yes
2-Methylpentane (107-83-5)	Yes	Yes	No	Yes
3-Methylpentane (96-14-0)	Yes	Yes	No	Yes
Pentane (109-66-0)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\--------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Hexane (110-54-3)	No	No	Yes	No
Methylcyclopentane (96-37-7)	No	No	No	No
2-Methylpentane (107-83-5)	No	No	No	No
3-Methylpentane (96-14-0)	No	No	No	No
Pentane (109-66-0)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\--------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Hexane (110-54-3)	5000	No	No
Methylcyclopentane (96-37-7)	No	No	No
2-Methylpentane (107-83-5)	No	No	No
3-Methylpentane (96-14-0)	No	No	No
Pentane (109-66-0)	No	No	Yes

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: Yes Pressure: No
Reactivity: No (Mixture / Liquid)

Australian Hazchem Code: 3[Y]E
Poison Schedule: No information found.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.
Label Precautions:
Keep away from heat, sparks and flame.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor or mist.
Avoid contact with eyes, skin and clothing.
Label First Aid:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. In all cases call a physician.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 8. Updated the ACGIH part of the Airborne section of TAB 8.
Disclaimer:
****

Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY,

ATTACHMENT 22.4

MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.

****

Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

ATTACHMENT 22.5

**** SECTION 1 - CHEMICAL PRODUCT AND COMPANY IDENTIFICATION ****

MSDS Name: Azulene, 99%
Catalog Numbers:
AC105180000, AC105180010, AC105181000
Synonyms:
Bicyclo[5.3.0]decapentaene; Cyclopentacycloheptene.
Company Identification (Europe): Acros Organics BVBA
Janssen Pharmaceuticalaan 3a
2440 Geel, Belgium
Company Identification (USA): Acros Organics
One Reagent Lane
Fairlawn, NJ 07410
For information in North America, call: 800-ACROS-01
For information in Europe, call: 0032(0) 14575211
For emergencies in the US, call CHEMTREC: 800-424-9300
For emergencies in Europe, call: 0032(0) 14575299

**** SECTION 2 - COMPOSITION, INFORMATION ON INGREDIENTS ****

CAS#	Chemical Name	%	EINECS#
275-51-4	Azulene	99.0	205-993-6

Hazard Symbols: XI
Risk Phrases: 36/37/38

**** SECTION 3 - HAZARDS IDENTIFICATION ****

EMERGENCY OVERVIEW
Irritating to eyes, respiratory system and skin.

Potential Health Effects
Eye:
Causes eye irritation. May cause chemical conjunctivitis.
Skin:
Causes skin irritation.
Ingestion:
May cause gastrointestinal irritation with nausea, vomiting and diarrhea.
Inhalation:
Causes respiratory tract irritation. Can produce delayed pulmonary edema.
Chronic:
Effects may be delayed.

**** SECTION 4 - FIRST AID MEASURES ****

Eyes:
Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Get medical aid.
Skin:
Get medical aid. Flush skin with plenty of soap and water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse.
Ingestion:
Never give anything by mouth to an unconscious person. Get medical aid. Do NOT induce vomiting. If conscious and alert, rinse mouth and drink 2-4 cupfuls of milk or water.
Inhalation:
Remove from exposure to fresh air immediately. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical aid.
Notes to Physician:
Treat symptomatically and supportively.

**** SECTION 5 - FIRE FIGHTING MEASURES ****

ATTACHMENT 22.5

General Information:
As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear. During a fire, irritating and highly toxic gases may be generated by thermal decomposition or combustion.
Extinguishing Media:
In case of fire, use water, dry chemical, chemical foam, or alcohol-resistant foam. Use water spray, dry chemical, carbon dioxide, or appropriate foam.

**** SECTION 6 - ACCIDENTAL RELEASE MEASURES ****

General Information: Use proper personal protective equipment as indicated in Section 8.
Spills/Leaks:
Clean up spills immediately, observing precautions in the Protective Equipment section. Sweep up or absorb material, then place into a suitable clean, dry, closed container for disposal. Provide ventilation.

**** SECTION 7 - HANDLING and STORAGE ****

Handling:
Wash thoroughly after handling. Remove contaminated clothing and wash before reuse. Avoid contact with eyes, skin, and clothing. Keep container tightly closed. Avoid ingestion and inhalation. Use with adequate ventilation. Wash clothing before reuse.
Storage:
Keep container closed when not in use. Store in a tightly closed container. Store in a cool, dry, well-ventilated area away from incompatible substances.

**** SECTION 8 - EXPOSURE CONTROLS, PERSONAL PROTECTION ****

Engineering Controls:
Facilities storing or utilizing this material should be equipped with an eyewash facility and a safety shower. Use adequate ventilation to keep airborne concentrations low.
Personal Protective Equipment

Eyes:
Wear safety glasses and chemical goggles if splashing is possible.
Skin:
Wear appropriate protective gloves and clothing to prevent skin exposure.
Clothing:
Wear appropriate protective clothing to prevent skin exposure.
Respirators:
A respiratory protection program that meets OSHA's 29 CFR :1910.134 and ANSI Z88.2 requirements or European Standard EN 149 must be followed whenever workplace conditions warrant a respirator's use. Wear a NIOSH/MSHA or European Standard EN 149 approved full-facepiece airline respirator in the positive pressure mode with emergency escape provisions.

**** SECTION 9 - PHYSICAL AND CHEMICAL PROPERTIES ****

Physical State:	Flakes
Appearance:	dark blue
Odor:	None reported.
pH:	Not available.
Vapor Pressure:	Not available.
Vapor Density:	Not available.
Evaporation Rate:	Not available.
Viscosity:	Not available.
Boiling Point:	242 deg C @ 760.00mmHg
Freezing/Melting Point:	99.00 - 100.50 deg C
Autoignition Temperature:	Not available.

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Flash Point: 270 deg C (518.00 deg F)
Explosion Limits, lower: Not available.
Explosion Limits, upper: Not available.
Decomposition Temperature: 270 deg C
Solubility: insoluble
Specific Gravity/Density:
Molecular Formula: C10H8
Molecular Weight: 128.17

**** SECTION 10 - STABILITY AND REACTIVITY ****

Chemical Stability:
Stable at room temperature in closed containers under normal storage and handling conditions.
Conditions to Avoid:
Incompatible materials, dust generation, excess heat, strong oxidants.
Incompatibilities with Other Materials:
Strong oxidizing agents.
Hazardous Decomposition Products:
Carbon monoxide, irritating and toxic fumes and gases, carbon dioxide.
Hazardous Polymerization: Has not been reported.

**** SECTION 11 - TOXICOLOGICAL INFORMATION ****

RTECS#:
CAS# 275-51-4: CO4570000
LD50/LC50:
CAS# 275-51-4: Oral, mouse: LD50 = >3 gm/kg; Oral, rat: LD50 = >4 gm/kg.
Carcinogenicity:
Azulene -
Not listed by ACGIH, IARC, NIOSH, NTP, or OSHA.
Epidemiology:
No information available.
Teratogenicity:
No information available.
Reproductive Effects:
No information available.
Neurotoxicity:
No information available.
Mutagenicity:
No information available.
Other Studies:
No data available.

**** SECTION 12 - ECOLOGICAL INFORMATION ****

Other
No information available.

**** SECTION 13 - DISPOSAL CONSIDERATIONS ****

Dispose of in a manner consistent with federal, state, and local regulations.

**** SECTION 14 - TRANSPORT INFORMATION ****

US DOT
No information available
Canadian TDG
No information available.

**** SECTION 15 - REGULATORY INFORMATION ****

US FEDERAL
TSCA
CAS# 275-51-4 is listed on the TSCA inventory.
This material does not contain any Class 2 Ozone depletors.
Clean Water Act:

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None of the chemicals in this product are listed as Hazardous Substances under the CWA.
None of the chemicals in this product are listed as Priority Pollutants under the CWA.
None of the chemicals in this product are listed as Toxic Pollutants under the CWA.

OSHA:

None of the chemicals in this product are considered highly hazardous by OSHA.

STATE

Azulene is not present on state lists from CA, PA, MN, MA, FL, or NJ.
California No Significant Risk Level:
None of the chemicals in this product are listed.

European/International Regulations

European Labeling in Accordance with EC Directives

Hazard Symbols: XI
Risk Phrases:
R 36/37/38 Irritating to eyes, respiratory system and skin.
Safety Phrases:
S 24/25 Avoid contact with skin and eyes.

WGK (Water Danger/Protection)

CAS# 275-51-4: No information available.

United Kingdom Occupational Exposure Limits

Canada

CAS# 275-51-4 is listed on Canada's DSL List.
CAS# 275-51-4 is not listed on Canada's Ingredient Disclosure List.

Exposure Limits

**** SECTION 16 - ADDITIONAL INFORMATION ****

MSDS Creation Date: 9/02/1997 Revision #3 Date: 8/02/2000

The information above is believed to be accurate and represents the best information currently available to us. However, we make no warranty of merchantability or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use. Users should make their own investigations to determine the suitability of the information for their particular purposes. In no way shall the company be liable for any claims, losses, or damages of any third party or for lost profits or any special, indirect, incidental, consequential or exemplary damages, howsoever arising, even if the company has been advised of the possibility of such damages.

ATTACHMENT 22.6

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS | J.T.Baker

24 Hour Emergency Telephone 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

TOLUENE

MSDS Number: T3913 --- *Effective Date: 11/17/99*

1. Product Identification

Synonyms: Methylbenzene; Toluol; Phenylmethane
CAS No.: 108-88-3
Molecular Weight: 92.14
Chemical Formula: C6H5-CH3
Product Codes:
J.T. Baker: 5375, 5584, 5809, 5812, 9336, 9351, 9364, 9456, 9457, 9459, 9460, 9462, 9466, 9472, 9476
Mallinckrodt: 4483, 8091, 8092, 8604, 8608, 8610, 8611, V560

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Toluene	108-88-3	100%	Yes

3. Hazards Identification

Emergency Overview

POISON! DANGER! HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED OR ABSORBED THROUGH SKIN. VAPOR HARMFUL. FLAMMABLE LIQUID AND VAPOR. MAY AFFECT LIVER, KIDNEYS, BLOOD SYSTEM, OR CENTRAL NERVOUS SYSTEM. CAUSES IRRITATION

ATTACHMENT 22.6

TO SKIN, EYES AND RESPIRATORY TRACT.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

-

Health Rating: 2 - Moderate
Flammability Rating: 3 - Severe (Flammable)
Reactivity Rating: 0 - None
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

-

Potential Health Effects

Inhalation:
Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Very high concentrations may cause unconsciousness and death.
Ingestion:
Swallowing may cause abdominal spasms and other symptoms that parallel over-exposure from inhalation. Aspiration of material into the lungs can cause chemical pneumonitis, which may be fatal.
Skin Contact:
Causes irritation. May be absorbed through skin.
Eye Contact:
Causes severe eye irritation with redness and pain.
Chronic Exposure:
Reports of chronic poisoning describe anemia, decreased blood cell count and bone marrow hypoplasia. Liver and kidney damage may occur. Repeated or prolonged contact has a defatting action, causing drying, redness, dermatitis. Exposure to toluene may affect the developing fetus.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or impaired liver or kidney function may be more susceptible to the effects of this substance. Alcoholic beverage consumption can enhance the toxic effects of this substance.

---

4. First Aid Measures

Inhalation:
If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. CALL A PHYSICIAN IMMEDIATELY.
Ingestion:
Aspiration hazard. If swallowed, DO NOT INDUCE VOMITING. Give large quantities

of water. Never give anything by mouth to an unconscious person. Get medical attention immediately. If vomiting occurs, keep head below hips to prevent aspiration into lungs.
Skin Contact:
In case of contact, immediately flush skin with plenty of soap and water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Call a physician immediately.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

---

5. Fire Fighting Measures

Fire:
Flash point: 7C (45F) CC
Autoignition temperature: 422C (792F)
Flammable limits in air % by volume:
lel: 3.3; uel: 19
Flammable liquid and vapor!
Dangerous fire hazard when exposed to heat or flame. Vapors can flow along surfaces to distant ignition source and flash back.
Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Contact with strong oxidizers may cause fire or explosion. Sensitive to static discharge.
Fire Extinguishing Media:
Dry chemical, foam or carbon dioxide. Water may be used to flush spills away from exposures and to dilute spills to non-flammable mixtures.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Water spray may be used to keep fire exposed containers cool.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB(R) solvent adsorbent is recommended for spills of this product.

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
Toluene:
- OSHA Permissible Exposure Limit (PEL):
200 ppm (TWA); 300 ppm (acceptable ceiling conc.); 500 ppm (maximum conc.).
- ACGIH Threshold Limit Value (TLV):
50 ppm (TWA) skin, A4 - Not Classifiable as a Human Carcinogen.

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, a half-face organic vapor respirator may be worn for up to ten times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. A full-face piece organic vapor respirator may be worn up to 50 times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. For emergencies or instances where the exposure levels are not known, use a full-face piece positive-pressure, air-supplied respirator.
WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.

Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

Appearance:
Clear, colorless liquid.
Odor:
Aromatic benzene-like.
Solubility:
0.05 gm/100gm water @ 20C (68F).
Specific Gravity:
0.86 @ 20C / 4 C
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100
Boiling Point:
111C (232F)
Melting Point:
-95C (-139F)
Vapor Density (Air=1):
3.14
Vapor Pressure (mm Hg):
22 @ 20C (68F)
Evaporation Rate (BuAc=1):
2.24

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Containers may burst when heated.
Hazardous Decomposition Products:
Carbon dioxide and carbon monoxide may form when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Heat, flame, strong oxidizers, nitric and sulfuric acids, chlorine, nitrogen tetraoxide; will attack some forms of plastics, rubber, coatings.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

11. Toxicological Information

Toxicological Data:
Oral rat LD50: 636 mg/kg; skin rabbit LD50: 14100 uL/kg; inhalation rat LC50: 49 gm/m3/4H; Irritation data: skin rabbit, 500 mg, Moderate; eye rabbit, 2 mg/24H, Severe. Investigated as a tumorigen, mutagen, reproductive effector.
Reproductive Toxicity:
Has shown some evidence of reproductive effects in laboratory animals.

ATTACHMENT 22.6

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Toluene (108-88-3)	No	No	3

12. Ecological Information

Environmental Fate:
When released into the soil, this material may evaporate to a moderate extent. When released into the soil, this material is expected to leach into groundwater. When released into the soil, this material may biodegrade to a moderate extent. When released into water, this material may evaporate to a moderate extent. When released into water, this material may biodegrade to a moderate extent. When released into the air, this material may be moderately degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material is expected to have a half-life of less than 1 day. This material is not expected to significantly bioaccumulate. This material has a log octanol-water partition coefficient of less than 3.0. Bioconcentration factor = 13.2 (eels).
Environmental Toxicity:
This material is expected to be toxic to aquatic life. The LC50/96-hour values for fish are between 10 and 100 mg/l.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: TOLUENE
Hazard Class: 3
UN/NA: UN1294
Packing Group: II
Information reported for product/size: 390LB

International (Water, I.M.O.)

Proper Shipping Name: TOLUENE

ATTACHMENT 22.6

Hazard Class: 3.2
UN/NA: UN1294
Packing Group: II
Information reported for product/size: 390LB

---

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Toluene (108-88-3)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Toluene (108-88-3)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\---------------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Toluene (108-88-3)	No	No	Yes	No

--------\Federal, State & International Regulations - Part 2\---------------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Toluene (108-88-3)	1000	U220	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: Yes
SARA 311/312: Acute: Yes Chronic: Yes Fire: Yes Pressure: No
Reactivity: No (Pure / Liquid)

WARNING:
THIS PRODUCT CONTAINS A CHEMICAL(S) KNOWN TO THE STATE OF CALIFORNIA TO CAUSE BIRTH DEFECTS OR OTHER REPRODUCTIVE HARM.

Australian Hazchem Code: 3[Y]E
Poison Schedule: S6
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **2** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
POISON! DANGER! HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF

INHALED OR ABSORBED THROUGH SKIN. VAPOR HARMFUL. FLAMMABLE LIQUID AND VAPOR. MAY AFFECT LIVER, KIDNEYS, BLOOD SYSTEM, OR CENTRAL NERVOUS SYSTEM. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT.

Label Precautions:

Keep away from heat, sparks and flame.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor.
Avoid contact with eyes, skin and clothing.

Label First Aid:

Aspiration hazard. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If vomiting occurs, keep head below hips to prevent aspiration into lungs. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. In all cases call a physician immediately.

Product Use:

Laboratory Reagent.

Revision Information:

No changes.

Disclaimer:

****

Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.

****

Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

ATTACHMENT 22.7

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

Silica Gel

MSDS Number: S1610 --- ***Effective Date: 08/20/98***

1. Product Identification

Synonyms: Kieselgel; Daiso Gel; Silica, Amorphous
CAS No.: 63231-67-4
Molecular Weight: Not available.
Chemical Formula: Not Available.
Product Codes: 3404, 3405, 3406

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Silica Gel	63231-67-4	99 - 100%	Yes

3. Hazards Identification

Emergency Overview

CAUTION! MAY CAUSE IRRITATION TO SKIN, EYES, AND RESPIRATORY TRACT.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 2 - Moderate

ATTACHMENT 22.7

Flammability Rating: 0 - None
Reactivity Rating: 0 - None
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES
Storage Color Code: Orange (General Storage)

Potential Health Effects

This product contains synthetic amorphous silica, not to be confused with crystalline silica such as quartz, cristobalite or tridymite or with diatomaceous earth or other naturally occurring forms of amorphous silica that frequently contain crystalline forms.

Inhalation:
May cause dryness and irritation to mucous membranes, nose, and throat. Symptoms may include coughing, sore throat, and wheezing.
Ingestion:
No adverse effects expected.
Skin Contact:
May cause irritation with dryness and abrasion.
Eye Contact:
May cause irritation, redness and pain.
Chronic Exposure:
Repeated exposure may cause symptoms similar to those listed for acute effects. Synthetic amorphous silica does not produce silicosis.
Aggravation of Pre-existing Conditions:
No information found.

4. First Aid Measures

Inhalation:
Remove to fresh air. Get medical attention for any breathing difficulty.
Ingestion:
Give several glasses of water to drink to dilute. If large amounts were swallowed, get medical advice.
Skin Contact:
Immediately flush skin with plenty of soap and water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention if irritation develops.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting upper and lower eyelids occasionally. Get medical attention if irritation persists.

5. Fire Fighting Measures

ATTACHMENT 22.7

Fire:
Not considered to be a fire hazard.
Explosion:
Not considered to be an explosion hazard.
Fire Extinguishing Media:
Use any means suitable for extinguishing surrounding fire.
Special Information:
Use protective clothing and breathing equipment appropriate for the surrounding fire.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Spills: Sweep up and containerize for reclamation or disposal. Vacuuming or wet sweeping may be used to avoid dust dispersal.

---

7. Handling and Storage

Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect against physical damage. When pouring into a container of flammable liquid, ground both containers electrically to prevent a static electric spark. Containers of this material may be hazardous when empty since they retain product residues (dust, solids); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
Silica (synthetic, amorphous):
- OSHA Permissible Exposure Limit (PEL) -
80/(%SiO2) mg/m3 (TWA) for amorphous silica, including natural diatomaceous earth.
- ACGIH Threshold Limit Value (TLV) -
10 mg/m3 (TWA) for amorphous precipitated silica and amorphous silica gel.
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, a half-face dust/mist respirator may be worn for up to ten times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. A full-face piece dust/mist respirator may be worn up to 50 times the exposure limit, or the maximum use concentration specified by the appropriate regulatory agency, or respirator

supplier, whichever is lowest. For emergencies or instances where the exposure levels are not known, use a full-facepiece positive-pressure, air-supplied respirator.
WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.
Skin Protection:
Wear protective gloves and clean body-covering clothing.
Eye Protection:
Use chemical safety goggles. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

Appearance:
White granular powder.
Odor:
Odorless.
Solubility:
Negligible (< 0.1%)
Specific Gravity:
2.10
pH:
3 - 8 (in 5% slurry)
% Volatiles by volume @ 21C (70F):
0
Boiling Point:
2230C (4046F)
Melting Point:
1610C (2930F)
Vapor Density (Air=1):
Not applicable.
Vapor Pressure (mm Hg):
Not applicable.
Evaporation Rate (BuAc=1):
No information found.

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products:
Oxides of carbon and silicon may be formed when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Reacts with hydrogen fluoride, fluorine, oxygen difluoride, chlorine trifluoride, strong acids, strong bases, and oxidizers.

Conditions to Avoid:
Moisture, extreme heat, and incompatibles.

11. Toxicological Information

No LD50/LC50 information found relating to normal routes of occupational exposure.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Silica Gel (63231-67-4)	No	No	None

12. Ecological Information

Environmental Fate:
When released into the soil, this material is not expected to biodegrade. When released into water, this material is not expected to biodegrade.
Environmental Toxicity:
This material is not expected to be toxic to aquatic life.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be managed in an appropriate and approved waste disposal facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Not regulated.

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\----------

Ingredient	TSCA	EC	Japan	Australia
Silica Gel (63231-67-4)	Yes	No	No	Yes

--------\Chemical Inventory Status - Part 2\----------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.

ATTACHMENT 22.7

Ingredient				
Silica Gel (63231-67-4)	Yes	Yes	No	Yes

\Federal, State & International Regulations - Part 1\

Ingredient	-SARA 302- RQ	TPQ	-SARA 313- List	Chemical Catg.
Silica Gel (63231-67-4)	No	No	No	No

\Federal, State & International Regulations - Part 2\

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Silica Gel (63231-67-4)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: No Pressure: No
Reactivity: No (Pure / Solid)

Australian Hazchem Code: No information found.
Poison Schedule: No information found.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

16. Other Information

NFPA Ratings: Health: **1** Flammability: **0** Reactivity: **0**
Label Hazard Warning:
CAUTION! MAY CAUSE IRRITATION TO SKIN, EYES, AND RESPIRATORY TRACT.
Label Precautions:
Avoid contact with eyes, skin and clothing.
Avoid breathing dust.
Keep container closed.
Use with adequate ventilation.
Wash thoroughly after handling.
Label First Aid:
If inhaled, remove to fresh air. Get medical attention for any breathing difficulty. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Get medical attention if irritation develops or persists.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 16.
Disclaimer:
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ATTACHMENT 22.7

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Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

ATTACHMENT 22.8

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS | J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

SODIUM SULFATE

MSDS Number: S5022 --- ***Effective Date: 11/17/99***

1. Product Identification

Synonyms: Sodium sulfate decahydrate; disodium sulfate decahydrate; glauber's salt; sulfuric acid, sodium salt, decahydrate; sodium sulfate, 10-hydrate
CAS No.: 7757-82-6 (Anhydrous)
Molecular Weight: 322.19
Chemical Formula: Na2SO4.10H2O
Product Codes:
J.T. Baker: 3889, 3890
Mallinckrodt: 8012, 8027

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Sodium Sulfate	7757-82-6	98 - 100%	Yes

3. Hazards Identification

Emergency Overview

As part of good industrial and personal hygiene and safety procedure, avoid all unnecessary exposure to the chemical substance and ensure prompt removal from skin, eyes and clothing.

ATTACHMENT 22.8

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 0 - None
Flammability Rating: 0 - None
Reactivity Rating: 0 - None
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT
Storage Color Code: Orange (General Storage)

Potential Health Effects

Inhalation:
Not expected to be a health hazard.
Ingestion:
Mildly toxic by ingestion. Systemic toxicity is unlikely unless massive amounts have been swallowed. Drinking water with > 500 mg/L may result in gastrointestinal irritation.
Skin Contact:
No adverse effects expected.
Eye Contact:
No adverse effects expected but dust may cause mechanical irritation.
Chronic Exposure:
No information found.
Aggravation of Pre-existing Conditions:
No information found.

4. First Aid Measures

Inhalation:
Not expected to require first aid measures.
Ingestion:
Give several glasses of water to drink to dilute. If large amounts were swallowed, get medical advice.
Skin Contact:
Wash exposed area with soap and water. Get medical advice if irritation develops.
Eye Contact:
Wash thoroughly with running water. Get medical advice if irritation develops.

5. Fire Fighting Measures

Fire:
Not considered to be a fire hazard.

Explosion:
Not considered an explosion hazard, but violent explosions occur when potassium sulfate and sodium sulfate are melted with aluminum.
Fire Extinguishing Media:
Use any means suitable for extinguishing surrounding fire.
Special Information:
Use protective clothing and breathing equipment appropriate for the surrounding fire.

6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Spills: Sweep up and containerize for reclamation or disposal. Vacuuming or wet sweeping may be used to avoid dust dispersal.

7. Handling and Storage

Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect against physical damage. Isolate from incompatible substances. Containers of this material may be hazardous when empty since they retain product residues (dust, solids); observe all warnings and precautions listed for the product.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
None established.
Ventilation System:
In general, dilution ventilation is a satisfactory health hazard control for this substance. However, if conditions of use create discomfort to the worker, a local exhaust system should be considered.
Personal Respirators (NIOSH Approved):
For conditions of use where exposure to the dust or mist is apparent, a half-face dust/mist respirator may be worn. For emergencies or instances where the exposure levels are not known, use a full-face positive-pressure, air-supplied respirator. WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.
Skin Protection:
Wear protective gloves and clean body-covering clothing.
Eye Protection:
Use chemical safety goggles. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

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Appearance:
White efflorescent crystals or granules.
Odor:
Odorless.
Solubility:
Soluble in 1.5 parts of water @ 25C (77F).
Specific Gravity:
1.46
pH:
Aqueous solution is neutral.
% Volatiles by volume @ 21C (70F):
0
Boiling Point:
No information found.
Melting Point:
32C (90F) Loses water at 100C (212F). Anhydrous melts ca. 844C (1551F)
Vapor Density (Air=1):
No information found.
Vapor Pressure (mm Hg):
No information found.
Evaporation Rate (BuAc=1):
No information found.

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10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Effloresces in dry air.
Hazardous Decomposition Products:
Oxides of sulfur and sodium may form when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
In combination with sodium sulfate, aluminum and magnesium will explode @ 800C (1472F); strong mineral acids and bases.
Conditions to Avoid:
Air, incompatibles.

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11. Toxicological Information

Investigated as a tumorigen, mutagen, reproductive effector. No LD50/LC50 information found relating to normal routes of occupational exposure.

--------\Carcer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category

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Sodium Sulfate (7757-82-6)	No	No	None

12. Ecological Information

Environmental Fate:
When released into the soil, this material is expected to leach into groundwater. This material is not expected to significantly bioaccumulate.
Environmental Toxicity:
This material is not expected to be toxic to aquatic life. The LC50/96-hour values for fish are over 100 mg/l. The EC50/48-hour values for daphnia are over 100 mg/l.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be managed in an appropriate and approved waste disposal facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Not regulated.

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Sodium Sulfate (7757-82-6)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Sodium Sulfate (7757-82-6)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\--------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Sodium Sulfate (7757-82-6)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\--------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Sodium Sulfate (7757-82-6)	No	No	No

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Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: No Chronic: No Fire: No Pressure: No
Reactivity: No (Mixture / Solid)

Australian Hazchem Code: No information found.
Poison Schedule: No information found.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

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16. Other Information

NFPA Ratings: Health: **1** Flammability: **0** Reactivity: **0**
Label Hazard Warning:
As part of good industrial and personal hygiene and safety procedure, avoid all unnecessary exposure to the chemical substance and ensure prompt removal from skin, eyes and clothing.
Label Precautions:
None.
Label First Aid:
Not applicable.
Product Use:
Laboratory Reagent.
Revision Information:
No changes.
Disclaimer:
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Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.
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ATTACHMENT 22.8

Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

DISTRIBUTION OF ORGANOCHLORINE PESTICIDES AND PCBs (AS AROCLORS) IN SILICA GEL COLUMN FRACTIONS

Compound	Fraction I	Fraction II
Hexachlorobutadiene	X	
1,2,4,5-Tetrachlorobenzene	X	
Hexachlorocyclopentadiene	X	
1,2,3,4-Tetrachlorobenzene	X	
Pentachlorobenzene	X	
alpha-BHC		X
Hexachlorobenzene	X	
beta-BHC		X
gamma-BHC (Lindane)		X
Pentachloronitrobenzene	<30%	>70%
delta-BHC		X
Heptachlor	X	
Aldrin	X	
Heptachlor Epoxide		X
gamma-Chlordane		X
alpha-Chlordane		X
Endosulfan I		X
Dieldrin		X
4-4'-DDE	>70%	<30%
Endrin		X
Endosulfan II		X
4,4,'-DDD		X
Endrin Aldehyde		X
Endosulfan Sulfate		X
4,4'-DDT	<20%	>80%
Endrin Ketone		X
Hexabromobenzene	X	
Methoxychlor		X
Mirex	X	
PBB (BP-6)	X	
Toxaphene		X
PCBS (as Aroclors)	X	
TCMX (surrogate)	X	
DCB (surrogate)	X	

Attachment 22.10

The Lancer dishwasher has two programs that we use. Program 5 uses deionized water and program 4 uses tap water.

Functions	Possibility	Our Program
PREWASH 1 PERIOD	0-30 MINUTES	1 MINUTE
FILLING	HOT/COLD	HOT
PREWASH 1 TEMPERATURE	0-95°C	50°C
PREWASH 1 DETERGENT	0-6 MINUTES	24 SECONDS
PREWASH 1 ADDITIVE 2 (ACID)	0-6 MINUTES	0
PREWASH 2 PERIOD	0-30 MINUTES	2 MINUTES
FILLING	HOT/COLD	HOT
PREWASH 2 TEMPERATURE	0-95°C	65°C
PREWASH 2 DETERGENT	0-6 MINUTES	24 SECONDS
PREWASH 2 ADDITIVE 2	0-6 MINUTES	0
PREWASH 3 PERIOD	0-30 MINUTES	0
FILLING	HOT/COLD	0
PREWASH 3 TEMPERATURE	0-95°C	0
PREWASH 3 DETERGENT	0-6 MINUTES	0
PREWASH 3 ADDITIVE 2	0-6 MINUTES	0
WASH PERIOD	0-30 MINUTES	4 MINUTES
FILLING	HOT/COLD	HOT
WASH TEMPERATURE	0-95°C	80°C
WASH DETERGENT	0-6 MINUTES	1 MINUTE. 6 SECONDS
WASH ADDITIVE 2	0-6 MINUTES	0
RUNNING WATER RINSE 1	0-9 MINUTES	2 MINUTES
ACID RINSE PERIOD	0-30 MINUTES	2 MINUTES
ACID	0-6 MINUTES	2 MINUTES
RUNNING WATER RINSE 2	0-9 MINUTES	7 MINUTES
COLD DEMI. WATER RINSE 1	0-30 MINUTES	0
COLD DEMI. WATER RINSE 2	0-30 MINUTES	0
COLD DEMI. WATER RINSE 3	0-30 MINUTES	0
COLD DEMI. WATER RINSE 4	0-3 MINUTES	0
HOT DEMI. WATER RINSE	0-30 MINUTES	2 MINUTES
HOT DEMI. TEMPERATURE	0-95°C	75°C
HOT DEMI. ADDITIVE	0-6 MINUTES	0
DRYING	0-90 MINUTES	25 MINUTES

DEQ

ENVIRONMENTAL LAB

MDEQ SOP # 403 REVISION #____

EFFECTIVE DATE 04/02

PAGE 1 OF 11 COPY____

RELEASED BY_____ DATE 6/13/02
COMPLIANCE OFFICER

FLORISIL CLEANUP

1.0 Applicable Analytical Methods

1.1 Florisil Cleanup, U.S. Environmental Protection Agency, Method 3620B, SW846, Revision 2, December 1996.

1.2 Quality Control, U.S. Environmental Protection Agency, Chapter 1, SW846, Revision 1, July 1992.

1.3 Organic Extraction and Sample Preparation, U.S. Environmental Protection Agency, Method 3500B, SW846, Revision 2, December 1996.

1.4 Cleanup, U.S. Environmental Protection Agency, Method 3600C, SW846, Revision 3, December 1996.

2.0 Matrix or Matrices

2.1 Aqueous.

2.2 Soil.

2.3 Sediment.

2.4 Sludge.

2.5 Clay.

2.6 Oil.

2.7 Solid or liquid waste.

3.0 Method Detection Limits (MDLs)

3.1 All MDLs are performed according to the Code of Federal Regulations 40 CFR, Part 136, Appendix B. See internal procedure SOP 103.

3.2 MDLs are extracted according to internal procedure SOP 405 and cleaned up according to internal procedure SOP 400 prior to florisil cleanup. The extract must be in hexane.

4.0 Scope and Application

4.1 This method provides for the cartridge cleanup of pesticide residues and other chlorinated hydrocarbons, the separation of nitrogen compounds from hydrocarbons, the separation of aromatic compounds from aliphatic-aromatic mixtures, and similar applications for use with fats, oils, ketones, glycerides, alkaloids, and some carbohydrates.

4.2 Florisil is a magnesium silicate with basic properties and can be used to separate analytes from interfering compounds prior to sample analysis by chromatographic methods.

4.3 This method includes procedures for cleanup of sample extracts containing the following analyte groups:

Phthalate esters	Chlorinated hydrocarbons
Nitrosamines	Organochlorine pesticides
Nitroaromatics	Organophosphates
Haloethers	Organophosphorus pesticides
Aniline and aniline derivatives	PCBs

5.0 Method Summary

5.1 Water samples are extracted by following internal procedure SOP 405 and are cleaned up as in internal procedure SOP 400. Solid samples are extracted by following internal procedure SOP 401 and are cleaned up as in internal procedure SOP 400. Oil samples are prepared as defined in internal procedure SOP 404 and are cleaned up as in internal procedure SOP 400. The sample extracts are then solvent exchanged to hexane.

5.2 Solid-phase extraction cartridges containing 40 µm particles of florisil (60 Å pores) are rinsed with appropriate solvents.

5.3 Sample extracts are introduced onto the top of the pre-packed column and eluted with suitable solvent(s). The eluate is concentrated to 1 mL in hexane and analyzed

by gas chromatography with electron capture detection (GC-ECD)(internal procedure SOP 502).

6.0 Definitions

6.1 Method Preparation Blank (MPB) - An aliquot of a blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and surrogates that are used with samples.

6.2 Laboratory Control Sample (LCS) - An aliquot of blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is treated exactly as a sample including extraction, cleanup, and analysis.

6.3 Matrix Spike (MS) - An aliquot of a sample to which known quantities of the target analytes are added in the laboratory. The MS is treated exactly as a sample including extraction, cleanup, and analysis. The background concentrations of the analytes in the sample must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

6.4 Matrix Spike Duplicate (MSD) - A second aliquot of sample to which known quantities of the target analytes are added in the laboratory. The MSD is treated exactly as a sample including extraction, cleanup, and analysis. The background concentrations of the analytes in the sample must be determined in a separate aliquot and the measured values in the MSD corrected for background concentrations. Analyses of the MS and MSD indicate precision associated with laboratory procedures.

7.0 Interferences

7.1 Interferences from contaminants can occur from solvents, glassware, and other sample processing equipment. Pesticide quality solvents (or better) and solvent rinsing of all glassware should help minimize contaminants.

7.2 The use of plastic must be minimized to prevent phthalate contamination.

7.3 Soap residue must be thoroughly rinsed from all glassware to avoid degradation of some pesticide compounds.

8.0 Safety

8.1 Safety glasses are required in all designated laboratory areas.

8.2 Be familiar with the Laboratory Chemical Hygiene Plan.

8.3 Be familiar with the Laboratory Safety Policy (internal procedure SOP 100).

8.4 Hexane, acetone, florisil and sodium sulfate material safety data sheets (MSDSs) are attached.

8.5 Nitrile or other solvent resistant gloves must be worn when handling solvents.

9.0 Equipment and Supplies

9.1 SPE PTFE manifold turn valves - Burdick & Jackson, part #9433T or equivalent.

9.2 SPE PTFE manifold port - Burdick & Jackson, part #9431 or equivalent.

9.3 Vials - 2 mL crimp vial with cap, Agilent, part #5181-3375 (vials), part #5181-1217 (caps) or equivalent.

9.3.1 Place 1 mL of hexane into each vial.
9.3.2 Mark the meniscus with a lab marker.
9.3.3 Dispose of the hexane. Let vial air dry.

9.4 Turbovap tubes - 50 mL capacity, Zymark, part #ZA2039 or equivalent.

9.5 Turbovap trays - Capacity of twelve 50 mL tubes.

9.6 Turbovap concentration workstation - With temperature control up to 95°C and nitrogen pressure from zero to 20 psi (settings: temperature = 35°C and pressure = 15 psi).

9.7 Pasteur pipets.

9.8 Bottle top dispensers - 1 to 10 mL.

9.9 Florisil manifold.

9.10 Graduated cylinders – 1000 mL.

10.0 Reagents and Standards

10.1 All reagents, solvents, and standards must be traceable to the stock inventory tracking log.

10.2 All reagents, solvents, and standards must be labeled with: date received, date opened, expiration date, tracking number, and receiver's initials.

10.3 All prepared reagents and standards must be labeled with: date prepared, expiration date, preparer's initials, tracking number, diluent, and description.

10.4 All standard logbooks must be completely filled out.

10.5 Nitrogen – Liquid.

10.6 Hexane (95% n-hexane) - Pesticide grade, demonstrated to be free of target analytes.

10.7 Acetone - HPLC grade, demonstrated to be free of target analytes.

10.8 Hexane/acetone (80/20).

10.8.1 Label an empty 4 L amber solvent bottle (Section 10.3).
10.8.2 Using a graduated cylinder, measure 3200 mL of hexane. Pour into a labeled amber bottle.
10.8.3 Using a graduated cylinder, measure 800 mL of acetone. Pour into a labeled amber bottle.
10.8.4 Mix by inverting the bottle several times. Vent.
10.8.5 Fill out the semivolatile standard logbook (Attachment 22.2).
10.8.6 Store the mixture in the water extractions florisil hood.

10.9 Florisil cartridges (2000 mg,15 mL) – Alltech, part #207962SP or equivalent.

10.10 Stock standards (SSs) – All standards must be certified reference materials. Manufacturer's certificates of analysis are retained by the DEQ laboratory and are kept as part of the overall quality assurance/quality control (QA/QC) requirements. All SSs must be given a unique lab tracking number and logged into the SS (Environmental Laboratory) tracking logbook (Attachment 22.1). Store the SSs in amber sealed ampules. Protect the standards from light. All standards are received in hexane unless otherwise noted.

10.10.1 SSs – SSs are purchased as mixes in sealed ampules and stored in a freezer at a temperature lower than 0°C.

10.10.1.1 Custom pesticide standard (1000 µg/mL) in hexane:toluene - Accustandard Inc., part #S-7179-R1 or equivalent.

Aldrin	Endosulfan Sulfate
alpha-BHC	Endrin
beta-BHC	Endrin Aldehyde
delta-BHC	Endrin Ketone
gamma-BHC (Lindane)	Heptaclor
alpha-Chlordane	Heptaclor Epoxide
gamma-Chlordane	Methoxyclor
4,4'-DDD	Mirex
4,4'-DDE	Hexabromobenzene
4,4'-DDT	PBB (BP-6)
Dieldrin	TCMX (surrogate)
Endosulfan I	DCB (surrogate)
Endosulfan II	

10.10.1.2 Custom volatile organic compound (VOC) standard (1000 µg/mL) in Acetone - Accustandard Inc., part #S-8258 or equivalent.

Pentachlorobenzene	Hexachlorobenzene
Pentachloronitrobenzene	Hexachlorobutadiene
1,2,3,4-Tetrachlorobenzene	Hexachlorocyclopentadiene
1,2,4,5-Tetrachlorobenzene	

10.11 Intermediate and working standards (WSs) – All prepared standards must be given a unique laboratory tracking number and logged into the semivolatile standards logbook (Attachment 22.2). Store prepared standards in labeled amber bottles in a freezer. Prepared standards are good for up to six months.

10.11.1 Pesticide WS (250 µg/L) – Place hexane in a 500 mL volumetric flask. Inject 125 µL of custom pesticide standard (Section 10.10.1.1) and 125 µL of custom VOC standard (Section 10.10.1.2) directly into the hexane in the flask. Bring to volume with hexane. Cap. Mix.

10.11.2 For a 125 ppb standard, make a 1:2 dilution of the 250 ppb standard in hexane.

11.0 Sample Collection, Preservation, and Handling

11.1 Aqueous samples – See internal procedure SOP 405.

11.2 Solid samples – See internal procedure SOP 401.

11.3 Waste samples – See internal procedure SOP 404.

11.4 Holding times – Sample extracts may be analyzed for up to 40 days from the extraction date.

11.5 Storage – Sample extracts must be stored in a freezer and protected from light in crimp cap vials until analysis.

12.0 Quality Control (QC)

12.1 All associated QC samples must be processed through this cleanup method along with the sample extracts.

12.2 Extracted sample batches

12.2.1 Extracted sample batches consist of a maximum of 20 samples, 1 MPB, 1 MS, 1 MSD, and 1 LCS (if a MS/MSD is not available, a laboratory control sample duplicate [LCSD] must be analyzed).

12.2.2 MSs and MSDs are actual samples that are selected at random from within the batch of 20.

12.2.3 All 20 samples and the QC samples are spiked with known surrogate concentrations. The recoveries for these compounds are used as indicators of possible problems that may occur during extraction or during cleanup of individual samples.

12.2.4 The MS, MSD, LCS, and LCSD are spiked at a known concentration with target analyte compounds. The replicate MSs or LCSs are used to evaluate the entire batch for precision and accuracy.

12.3 Initial demonstration of ability (IDOA) - Each analyst must demonstrate initial proficiency with this method by generating data of acceptable accuracy and precision for the target analytes. This is accomplished by extraction and cleanup of four aliquots of a reference sample.

12.3.1 Five aliquots of reagent water or sand are extracted using internal procedure SOP 401 or internal procedure SOP 405. One of these is a MPB, and the other four are spiked at a mid-range concentration with all the analytes of interest.
12.3.2 Sample extracts are taken through all the normal sample cleanup methods.
12.3.3 Extracts are then analyzed by GC-ECD. See internal procedure SOP 502 for further details.

13.0 Calibration and Standardization

13.1 Each new lot of florisil is tested to determine if compound recoveries are sufficient and if cartridges are free of interferences. The procedure is as follows:

13.1.1 Prepare hexane/acetone mix (Section 10.8).
13.1.2 Attach turn valves and manifold ports to three cartridges. Place the assembled cartridges on the manifold.
13.1.3 Add 5 mL of 80/20 hexane/acetone to each cartridge. Place waste containers under the cartridges.
13.1.4 Open the valves enough to allow several drops to pass through each column. Close the valves. Allow the cartridges to soak for at least five minutes.
13.1.5 Open the valves. Drain. Do net let the cartridges go dry. Close the valves.
13.1.6 Add 5 mL of hexane to each cartridge.
13.1.7 Open the valves. Drain. Do not let the cartridges go dry. Close the valves.
13.1.8 Label the cartridges (MPB, LCS1, and LCS2).
13.1.9 Add 1 mL of hexane to the cartridge labeled MPB. Add 1 mL of 125 ppb pesticide WS (Section 10.11.2) to each LCS cartridge.
13.1.10 Open the valve to drain into the waste container. When the entire extract has passed through, but before it goes dry, close the valve.
13.1.11 Place a labeled turbovap tube under each cartridge
13.1.12 Elute each cartridge with 9 mL of hexane/acetone (80/20). Open the valves. Let the cartridges go dry.
13.1.13 Concentrate each sample on the turbovap workstation to less than 1 mL.
13.1.14 Transfer each sample to a 1 mL pre-marked crimp cap vial (Section 9.3) using pasteur pipets. Dilute to 1 mL with hexane. Cap.
13.1.15 Label each vial (MPB, LCS1, and LCS2).
13.1.16 Fill out the solvent/reagent checking logbook (Attachment 22.7).
13.1.17 Fill out the florisil logbook. Copy onto yellow colored paper (Attachment 22.6).
13.1.18 Give the yellow copy of the florisil logbook page and the samples to the analyst.

14.0 Procedure

14.1 Sample preparation

14.1.1 Extracts must be in hexane.
14.1.2 Extracts must be taken off the GPC. The solvent must be exchanged prior to being florisilied.
14.1.3 Concentrate extracts to approximately 1 mL in hexane in a turbovap tube.

14.2 Cartridge preparation

14.2.1 Attach a SPE PTFE turn valve to the tip of each florisil cartridge.
14.2.2 Attach a SPE PTFE manifold port to the tip of each turn valve.
14.2.3 Place assembled florisil cartridges in the manifold apparatus. Place waste containers under the cartridges.
14.2.4 Add 5 mL of 80/20 hexane/acetone (Section 10.8) to each cartridge. Open the valves. Allow several drops of solvent to pass through each column. Close the valves. Allow the cartridges to soak for at least five minutes.
14.2.5 Open the valves. Drain. Do not let the cartridges go dry. Close the valves.
14.2.6 Add 5 mL of hexane to each cartridge.
14.2.7 Open the valves. Drain. Do not let the cartridges go dry. Close the valves.
14.2.8 Label the cartridges.
14.2.9 Immediately proceed to Section 14.3.1.

14.3 Procedure

14.3.1 Transfer the sample from the turbovap tube with a pasteur pipet to the appropriately labeled cartridge.
14.3.2 Open the valve to drain into the waste container. When the entire extract has passed through, but before it goes dry, close the valves.
14.3.3 Rinse the turbovap tube with 1 mL of hexane. Transfer the rinsate to the cartridge.
14.3.4 Place the same turbovap tube under the cartridge. Drain. Do not allow it to go dry.
14.3.5 Elute each cartridge with 9 mL of hexane/acetone (80/20). Let the cartridge go dry.
14.3.6 Concentrate each sample on the turbovap workstation to less than 1 mL.
14.3.7 Transfer each sample to a 1 mL pre-marked crimp cap vial (Section 9.3) using pasteur pipets. Dilute to 1 mL with hexane. Cap.
14.3.8 Label each vial with the sample number.
14.3.9 Fill out the florisil logbook. Copy onto yellow colored paper (Attachment 22.6).
14.3.10 Staple the yellow logbook copy to the batch sheets. Give this and the extracts to the analyst.

15.0 Calculations

15.1 See internal procedure SOP 502.

16.0 Method Performance

16.1 Each MDL study must be labeled with the analyst's name and the date.

16.2 Each IDOA study must be labeled with the analyst's name and the date. A copy must be placed into the analyst's training file.

17.0 Pollution Prevention

17.1 This method uses small quantities of organic solvents. The waste that is produced is collected for disposal.

17.2 For information concerning pollution prevention that may be applicable to laboratory operations, consult Less is Better: Laboratory Chemical Management for Waste Reduction available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, DC 20036.

18.0 Data Assessment

18.1 Florisil lot check (Section 13.1) – All recoveries must be 80 – 110% with no interfering peaks.

19.0 Corrective Actions

19.1 QA/QC failure – If the QA/QC in internal procedure SOP 502 is not met and if it is determined to be attributed to the extraction or cleanup, the samples may be repeated through this cleanup process.

20.0 Waste Management

20.1 The Laboratory Safety Officer arranges for proper waste disposal.

21.0 References

21.1 United States Code of Federal Regulations, Title 40, Part 136, Appendix A and B, U.S. Government Printing Office, Washington, DC.

21.2 Test Methods for Evaluating Solid Waste, U.S. Environmental Protection Agency, Document SW-846, Third Edition 1986, Final Update III, December 1996.

22.0 Attachments

22.1 Stock standard logbook page.

22.2 Semivolatile standard logbook page.

22.3 Hexane MSDS.

22.4 Acetone MSDS.

22.5 Florisil MSDS.

22.6 Florisil logbook page.

22.7 Solvent/reagent checking logbook page.

MDEQ SOP# 403 REVISION #____

APPROVED BY _____ DATE 5/17/02
CHIEF, LABORATORY SERVICES SECTION

APPROVED BY _____ DATE 5/16/02
COMPLIANCE OFFICER

APPROVED BY _____ DATE 4/23/02
QA OFFICER

APPROVED BY Carol Smith DATE 4-18-02
UNIT MANAGER

APPROVED BY _____ DATE 4/17/02
ANALYST

MDEQ-ENVIRONMENTAL LAB
STOCK TRACKING LOG

Environmental Lab Tracking Number	QTY (each)	Filley Tracking Number	Date Received (at lab)	Receiver Initials	Chemical,Analyte, Reagent or Solvent	Vendor and Catalog #	PO #	Manufacturer and Lot Number	Expiration Date
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
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FILE:ST-STD
12/30/98

MDEQ-ENVIRONMENTAL LAB
SEMI-VOLATILE ORGANIC STANDARDS

SVOC Tracking Number	Date Prepared	EL or SVOC Tracking Number	Analyte or Mix	Preparers Initials	Solvent	Solvent Tracking Number	Initial Concentration*	Initial Volume/Wt Standard*	Final Volume*	Final Concentration*	Expiration Date
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
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SE-											
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SE-											
SE-											
SE-											
SE-											
SE-											
SE-											

ATTACHMENT 22-2

FILE:SV-STD
12/30/98

*INCLUDE UNITS

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

HEXANES

MSDS Number: H2379 --- *Effective Date: 11/02/01*

1. Product Identification

Synonyms: Normal Hexane; Hexyl Hydride
CAS No.: 110-54-3 (n-hexane)
Molecular Weight: 86.18
Chemical Formula: CH3(CH2)4CH3 n-hexane
9277, 9306, 9309, N169

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Hexane	110-54-3	> 95%	Yes
Methylcyclopentane	96-37-7	1 - 2%	Yes
2-Methylpentane	107-83-5	1 - 2%	No
3-Methylpentane	96-14-0	1 - 2%	No
Pentane	109-66-0	0 - 2%	No

3. Hazards Identification

Emergency Overview

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF

INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Life)
Flammability Rating: 4 - Extreme (Flammable)
Reactivity Rating: 2 - Moderate
Contact Rating: 2 - Moderate
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

The health hazards addressed are for the major component: n-hexane.

Inhalation:
Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Greater exposure may cause muscle weakness, numbness of the extremities, unconsciousness and death.
Ingestion:
May produce abdominal pain, nausea. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms expected to parallel inhalation.
Skin Contact:
May cause redness, irritation, with dryness, cracking.
Eye Contact:
Vapors may cause irritation. Splashes may cause redness and pain.
Chronic Exposure:
Repeated or prolonged skin contact may defat the skin and produce irritation and dermatitis. Chronic inhalation may cause peripheral nerve disorders and central nervous system effects.

Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance. May affect the developing fetus.

---

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:
Aspiration hazard. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:

Remove any contaminated clothing. Wipe off excess from skin. Wash skin with soap and water for at least 15 minutes. Get medical attention if irritation develops or persists.

Eye Contact:

Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

Note to Physician:

BEI=2,5-hexadione in urine, sample at end of shift at workweeks end, 5 mg/g creatine. Also, measure n-hexane in expired air. Analgesics may be necessary for pain management, there is no specific antidote. Monitor arterial blood gases in cases of severe aspiration.

---

5. Fire Fighting Measures

Fire:

Flash point: -21C (-6F) CC
Autoignition temperature: 224C (435F)
Flammable limits in air % by volume:
lel: 1.1; uel: 7.5
Extremely Flammable Liquid and Vapor! Vapor may cause flash fire. Dangerous fire hazard when exposed to heat or flame.

Explosion:

Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Contact with oxidizing materials may cause extremely violent combustion.Explodes when mixed @ 28C with dinitrogen tetraoxide. Sensitive to static discharge.

Fire Extinguishing Media:

Dry chemical, foam or carbon dioxide. Water may be ineffective.

Special Information:

In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Water spray may be used to keep fire exposed containers cool. Vapors can flow along surfaces to distant ignition source and flash back. Vapor explosion hazard exists indoors, outdoors, or in sewers.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB® solvent adsorbent is recommended for spills of this product.

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from direct sunlight and any area where the fire hazard may be acute. Store in tightly closed containers (preferably under nitrogen atmosphere). Outside or detached storage is preferred. Inside storage should be in a standard flammable liquids storage room or cabinet. Separate from oxidizing materials. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
N-Hexane [110-54-3]:
-OSHA Permissible Exposure Limit (PEL): 500 ppm (TWA)
-ACGIH Threshold Limit Value (TLV): 50 ppm (TWA), Skin
other isomers of hexane
-ACGIH Threshold Limit Value (TLV): 500 ppm (TWA),1000ppm (STEL)
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded and engineering controls are not feasible, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. Breathing air quality must meet the requirements of the OSHA respiratory protection standard (29CFR1910.134).
Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.
Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

The following properties are for n-hexane, the major component.

Appearance:
Clear, colorless liquid.
Odor:
Gasoline-like odor.
Solubility:
Insoluble in water.
Specific Gravity:
0.66
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100
Boiling Point:
ca. 68C (ca. 154F)
Melting Point:
ca. -95C (ca. -139F)
Vapor Density (Air=1):
3.0
Vapor Pressure (mm Hg):
150 @ 25C (77F)
Evaporation Rate (BuAc=1):
9

---

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Heat will contribute to instability.
Hazardous Decomposition Products:
May produce acrid smoke and irritating fumes when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Strong oxidizers.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

---

11. Toxicological Information

N-Hexane: Oral rat LD50: 28710 mg/kg. Irritation eye rabbit: 10 mg mild. Investigated as a tumorigen, mutagen and reproductive effector.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category

Hexane (110-54-3)	No	No	None
Methylcyclopentane (96-37-7)	No	No	None
2-Methylpentane (107-83-5)	No	No	None
3-Methylpentane (96-14-0)	No	No	None
Pentane (109-66-0)	No	No	None

12. Ecological Information

Environmental Fate:
When released into the soil, this material may biodegrade to a moderate extent. When released into the soil, this material is not expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material may biodegrade to a moderate extent. When released to water, this material is expected to quickly evaporate. When released into the water, this material is expected to have a half-life between 1 and 10 days. This material has an estimated bioconcentration factor (BCF) of less than 100. This material has a log octanol-water partition coefficient of greater than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material is expected to be readily degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material is expected to have a half-life between 1 and 10 days.
Environmental Toxicity:
No information found.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: HEXANES
Hazard Class: 3
UN/NA: UN1208
Packing Group: II
Information reported for product/size: 215L

International (Water, I.M.O.)

Proper Shipping Name: HEXANES
Hazard Class: 3
UN/NA: UN1208
Packing Group: II
Information reported for product/size: 215L

---

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Hexane (110-54-3)	Yes	Yes	Yes	Yes
Methylcyclopentane (96-37-7)	Yes	Yes	No	Yes
2-Methylpentane (107-83-5)	Yes	Yes	Yes	Yes
3-Methylpentane (96-14-0)	Yes	Yes	Yes	Yes
Pentane (109-66-0)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Hexane (110-54-3)	Yes	Yes	No	Yes
Methylcyclopentane (96-37-7)	Yes	Yes	No	Yes
2-Methylpentane (107-83-5)	Yes	Yes	No	Yes
3-Methylpentane (96-14-0)	Yes	Yes	No	Yes
Pentane (109-66-0)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\--------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Hexane (110-54-3)	No	No	Yes	No
Methylcyclopentane (96-37-7)	No	No	No	No
2-Methylpentane (107-83-5)	No	No	No	No
3-Methylpentane (96-14-0)	No	No	No	No
Pentane (109-66-0)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\--------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Hexane (110-54-3)	5000	No	No
Methylcyclopentane (96-37-7)	No	No	No
2-Methylpentane (107-83-5)	No	No	No
3-Methylpentane (96-14-0)	No	No	No
Pentane (109-66-0)	No	No	Yes

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: Yes Pressure: No
Reactivity: No (Mixture / Liquid)

Australian Hazchem Code: 3[Y]E

Poison Schedule: No information found.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.
Label Precautions:
Keep away from heat, sparks and flame.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor or mist.
Avoid contact with eyes, skin and clothing.
Label First Aid:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. In all cases call a physician.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 8.
Disclaimer:
****

Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.
****

Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

ACETONE

MSDS Number: A0446 --- *Effective Date: 04/10/01*

1. Product Identification

Synonyms: Dimethylketone; 2-propanone; dimethylketal
CAS No.: 67-64-1
Molecular Weight: 58.08
Chemical Formula: (CH3)2CO
Product Codes:
J.T. Baker: 5356, 5580, 5805, 9001, 9002, 9003, 9004, 9005, 9006, 9007, 9008, 9009, 9010, 9015, 9036, 9125, 9254, 9271, A134, V655
Mallinckrodt: 0018, 2432, 2435, 2437, 2438, 2440, 2443, 2445, 2850, H451, H580, H981

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Acetone	67-64-1	99 - 100%	Yes

3. Hazards Identification

Emergency Overview

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL IF SWALLOWED OR INHALED. CAUSES

IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 1 - Slight
Flammability Rating: 4 - Extreme (Flammable)
Reactivity Rating: 2 - Moderate
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

Inhalation:
Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache. Higher concentrations can produce central nervous system depression, narcosis, and unconsciousness.
Ingestion:
Swallowing small amounts is not likely to produce harmful effects. Ingestion of larger amounts may produce abdominal pain, nausea and vomiting. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms are expected to parallel inhalation.
Skin Contact:
Irritating due to defatting action on skin. Causes redness, pain, drying and cracking of the skin.
Eye Contact:
Vapors are irritating to the eyes. Splashes may cause severe irritation, with stinging, tearing, redness and pain.
Chronic Exposure:
Prolonged or repeated skin contact may produce severe irritation or dermatitis.
Aggravation of Pre-existing Conditions:
Use of alcoholic beverages enhances toxic effects. Exposure may increase the toxic potential of chlorinated hydrocarbons, such as chloroform, trichloroethane.

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.
Ingestion:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately.
Skin Contact:

Immediately flush skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.

Eye Contact:

Immediately flush eyes with plenty of water for at least 15 minutes, lifting upper and lower eyelids occasionally. Get medical attention.

---

5. Fire Fighting Measures

Fire:

Flash point: -20C (-4F) CC
Autoignition temperature: 465C (869F)
Flammable limits in air % by volume:
lel: 2.5; uel: 12.8
Extremely Flammable Liquid and Vapor! Vapor may cause flash fire.

Explosion:

Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Vapors can flow along surfaces to distant ignition source and flash back. Contact with strong oxidizers may cause fire. Sealed containers may rupture when heated. This material may produce a floating fire hazard. Sensitive to static discharge.

Fire Extinguishing Media:

Dry chemical, alcohol foam or carbon dioxide. Water may be ineffective. Water spray may be used to keep fire exposed containers cool, dilute spills to nonflammable mixtures, protect personnel attempting to stop leak and disperse vapors.

Special Information:

In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB(R) solvent adsorbent is recommended for spills of this product.

---

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
Acetone:
-OSHA Permissible Exposure Limit (PEL):
1000 ppm (TWA)

-ACGIH Threshold Limit Value (TLV):
500 ppm (TWA), 750 ppm (STEL) A4 - not classifiable as a human carcinogen
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, a half-face organic vapor respirator may be worn for up to ten times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. A full-face piece organic vapor respirator may be worn up to 50 times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. For emergencies or instances where the exposure levels are not known, use a full-face piece positive-pressure, air-supplied respirator. WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.
Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.
Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

Appearance:
Clear, colorless, volatile liquid.

Odor:
Fragrant, mint-like
Solubility:
Miscible in all proportions in water.
Specific Gravity:
0.79 @ 20C/4C
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100
Boiling Point:
56.5C (133F) @ 760 mm Hg
Melting Point:
-95C (-139F)
Vapor Density (Air=1):
2.0
Vapor Pressure (mm Hg):
400 @ 39.5C (104F)
Evaporation Rate (BuAc=1):
ca. 7.7

---

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products:
Carbon dioxide and carbon monoxide may form when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Concentrated nitric and sulfuric acid mixtures, oxidizing materials, chloroform, alkalis, chlorine compounds, acids, potassium t-butoxide.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

---

11. Toxicological Information

Oral rat LD50: 5800 mg/kg; Inhalation rat LC50: 50,100mg/m3; Irritation eye rabbit, Standard Draize, 20 mg severe; investigated as a tumorigen, mutagen, reproductive effector.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Acetone (67-64-1)	No	No	None

12. Ecological Information

Environmental Fate:
When released into the soil, this material is expected to readily biodegrade. When released into the soil, this material is expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material is expected to readily biodegrade. When released to water, this material is expected to quickly evaporate. This material has a log octanol-water partition coefficient of less than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material may be moderately degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material may be moderately degraded by photolysis. When released into the air, this material is expected to be readily removed from the atmosphere by wet deposition.
Environmental Toxicity:
This material is not expected to be toxic to aquatic life. The LC50/96-hour values for fish are over 100 mg/l.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: ACETONE
Hazard Class: 3
UN/NA: UN1090
Packing Group: II
Information reported for product/size: 350LB

International (Water, I.M.O.)

Proper Shipping Name: ACETONE
Hazard Class: 3
UN/NA: UN1090
Packing Group: II
Information reported for product/size: 350LB

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Acetone (67-64-1)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Acetone (67-64-1)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\----------------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Acetone (67-64-1)	No	No	Yes	No

--------\Federal, State & International Regulations - Part 2\----------------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Acetone (67-64-1)	5000	U002	No

Chemical Weapons Convention: No TSCA 12(b): Yes CDTA: Yes
SARA 311/312: Acute: Yes Chronic: No Fire: Yes Pressure: No
Reactivity: No (Pure / Liquid)

Australian Hazchem Code: 2[Y]E
Poison Schedule: No information found.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL IF SWALLOWED OR INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM.
Label Precautions:
Keep away from heat, sparks and flame.
Keep container closed.

Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor.
Avoid contact with eyes, skin and clothing.

Label First Aid:

Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. In all cases, get medical attention.

Product Use:

Laboratory Reagent.

Revision Information:

No changes.

Disclaimer:

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****

Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS | J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

FLORISIL®

MSDS Number: F2546 --- *Effective Date: 11/02/01*

1. Product Identification

Synonyms: Synthetic magnesia-silica gel; Activated magnesium silicate; Florisil® 60-100 mesh; Florisil® 100-200 mesh; Florisil® 60-100 suitable for pesticide residue analysis; Magnesia-Silica Gel; Magnesium Silicate; Silicic Acid, Magnesium Salt
CAS No.: 1343-88-0
Molecular Weight: Not applicable to mixtures.
Chemical Formula: MgO 3.75 SiO2.xH2O
Product Codes: J.T. Baker: 3372, M368, M369
Mallinckrodt: 6875, 6876, 6877

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Synthetic Magnesia-silica gel	1343-88-0	90 - 100%	Yes

Florisil® is a registered trademark of U.S. Silica Company

3. Hazards Identification

Emergency Overview

CAUTION! MAY CAUSE IRRITATION TO SKIN, EYES, AND RESPIRATORY

TRACT.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 1 - Slight
Flammability Rating: 0 - None
Reactivity Rating: 0 - None
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT
Storage Color Code: Orange (General Storage)

Potential Health Effects

Inhalation:
May cause dryness and irritation to mucous membranes, nose, and throat.
Ingestion:
No adverse effects expected.
Skin Contact:
May cause irritation with dryness and abrasion.
Eye Contact:
No adverse effects expected but dust may cause mechanical irritation.
Chronic Exposure:
Grossly excessive and chronic inhalation of the dust may cause a progressive chemical pneumonitis, cyanosis, and pulmonary edema. Repeated or prolonged ingestion may contribute to the production of urinary calculi.
Aggravation of Pre-existing Conditions:
No information found.

---

4. First Aid Measures

Inhalation:
Remove to fresh air. Get medical attention for any breathing difficulty.
Ingestion:
Give several glasses of water to drink to dilute. If large amounts were swallowed, get medical advice.
Skin Contact:
Wash exposed area with soap and water. Get medical advice if irritation develops.
Eye Contact:
Wash thoroughly with running water. Get medical advice if irritation develops.

---

5. Fire Fighting Measures

Fire:
Not considered to be a fire hazard.

Explosion:
Not considered to be an explosion hazard.
Fire Extinguishing Media:
Use any means suitable for extinguishing surrounding fire.
Special Information:
Use protective clothing and breathing equipment appropriate for the surrounding fire.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Spills: Sweep up and containerize for reclamation or disposal. Vacuuming or wet sweeping may be used to avoid dust dispersal.

---

7. Handling and Storage

Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect against physical damage. Isolate from incompatible substances. Containers of this material may be hazardous when empty since they retain product residues (dust, solids); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
- OSHA Permissible Exposure Limit (PEL):
15 mg/m3 total dust, 5 mg/m3 respirable fraction for nuisance dusts.
- ACGIH Threshold Limit Value (TLV):
10 mg/m3 total dust containing no asbestos and < 1% crystalline silica for Particulates Not Otherwise Classified (PNOC).
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures as low as possible. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded and engineering controls are not feasible, a half facepiece particulate respirator (NIOSH type N95 or better filters) may be worn for up to ten times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest.. A full-face piece particulate respirator (NIOSH type N100 filters) may be worn up to 50 times the exposure limit, or the maximum use concentration specified by the appropriate regulatory agency, or respirator supplier, whichever is lowest. If oil particles (e.g. lubricants, cutting fluids, glycerine, etc.) are present, use a NIOSH type R or P filter. For emergencies or instances where the exposure

levels are not known, use a full-facepiece positive-pressure, air-supplied respirator. WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.
Skin Protection:
Wear protective gloves and clean body-covering clothing.
Eye Protection:
Use chemical safety goggles. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

Appearance:
White powder or granules.
Odor:
Odorless.
Solubility:
Insoluble in water.
Specific Gravity:
2.51
pH:
No information found.
% Volatiles by volume @ 21C (70F):
0
Boiling Point:
No information found.
Melting Point:
No information found.
Vapor Density (Air=1):
No information found.
Vapor Pressure (mm Hg):
No information found.
Evaporation Rate (BuAc=1):
No information found.

---

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products:
No information found.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Hydrofluoric acid will dissolve substance and produce corrosive silicon tetrafluoride.
Conditions to Avoid:
Dusting and incompatibles.

11. Toxicological Information

No LD50/LC50 information found relating to normal routes of occupational exposure.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Synthetic Magnesia-silica gel (1343-88-0)	No	No	None

12. Ecological Information

Environmental Fate:
No information found.
Environmental Toxicity:
No information found.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be managed in an appropriate and approved waste disposal facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Not regulated.

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\----------

Ingredient	TSCA	EC	Japan	Australia
Synthetic Magnesia-silica gel (1343-88-0)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\----------

	--Canada--

Ingredient	Korea	DSL	NDSL	Phil.
Synthetic Magnesia-silica gel (1343-88-0)	Yes	Yes	No	Yes

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Synthetic Magnesia-silica gel (1343-88-0)	No	No	No	No

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Synthetic Magnesia-silica gel (1343-88-0)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: No Fire: No Pressure: No
Reactivity: No (Pure / Solid)

Australian Hazchem Code: No information found.
Poison Schedule: No information found.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **1** Flammability: **0** Reactivity: **0**
Label Hazard Warning:
CAUTION! MAY CAUSE IRRITATION TO SKIN, EYES, AND RESPIRATORY TRACT.
Label Precautions:
Avoid breathing dust.
Avoid contact with eyes, skin and clothing.
Keep container closed.
Use with adequate ventilation.
Wash thoroughly after handling.
Label First Aid:
If inhaled, remove to fresh air. Get medical attention for any breathing difficulty. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Get medical attention if irritation develops or persists.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 8.
Disclaimer:

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Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

MDEQ-Environmental Lab
Florisil Cleanup Logbook-Organics

Scan:_____
Method: 3620
Date:_____
Analyst:_____
Batch #_____

ATTACHMENT 22.6

	Florsil Cartridge EL#		Hexane EL#		Hexane/Acetone SE#	
#	Sample #	Fraction #	Extraction Batch #	Volume of Extract Cleaned (uL)	Final Volume (uL)	Comments
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						

COMMENTS

MDEQ-ENVIRONMENTAL LAB
SOLVENT CHECKING LOG

Solvent	Manufacturer	Lot #	Date Checked	Analyst	Comments/ Results	Acceptable Yes/No

ATTACHMENT 22.7

FILE:SOL
01/09/99

ENVIRONMENTAL LAB

MDEQ SOP # 405 REVISION # 2

EFFECTIVE DATE 01/05

PAGE 1 OF 21 COPY

RELEASED BY _____ DATE 1/24/05
COMPLIANCE OFFICER

SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION

1.0 Applicable Analytical Methods

1.1 Organic Extraction and Sample Preparation, U.S. Environmental Protection Agency, Method 3500B, SW846, Revised, December 1996.

1.2 Separatory Funnel Liquid-Liquid Extraction, U.S. Environmental Protection Agency, Method 3510C, SW846, Revision 3, December 1996.

1.3 Organochlorine Pesticides and PCBs, U.S. Environmental Protection Agency, Method 608, 40 CFR, Part 136, Appendix A.

1.4 Chlorinated Hydrocarbons, U.S. Environmental Protection Agency, Method 612, 40 CFR, Part 136, Appendix A.

1.5 Base Neutrals and Acids, U.S. Environmental Protection Agency, Method 625, 40 CFR, Part 136, Appendix A.

1.6 Quality Control, U.S. Environmental Protection Agency, Chapter 1, SW846, Revision 1, July 1992.

2.0 Matrix or Matrices

2.1 Aqueous.

3.0 Method Detection Limits

3.1 All method detection limits (MDLs) shall be performed according to the Code of Federal Regulations, 40 CFR, Part 136, Appendix B. See internal procedure SOP 103.

3.2 The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. MDLs are analyzed annually or whenever major instrument maintenance occurs. MDLs should be calculated as in 40 CFR, Part 136, Appendix B.

3.3 A minimum of seven replicate extractions are performed using reagent water spiked with the analytes of interest at a level which meets the criteria in SOP 103.

3.3.1 One method preparation blank (MPB) is extracted with every set of MDLs.

3.3.2 The MPB and all the replicates must be extracted as if they were samples and must undergo the same cleanups as a sample.

4.0 Scope and Application

4.1 The method is used in the extraction of semi-volatile organic compounds from municipal and industrial discharges, surface waters, and groundwaters.

4.2 This method is designated to meet the monitoring requirements of the National Pollutant Discharge Elimination System and similar environmental programs.

4.3 This method is applicable to the extraction of semi-volatile organic compounds, halogenated hydrocarbons, polychlorinated biphenyls (PCBs), and certain organochlorine pesticides. See DEQ Laboratory SOP # 500 and SOP #502 for complete lists of compounds covered by this method.

5.0 Method Summary

5.1 A 1 L sample is serially extracted with methylene chloride (MeCl) using separatory funnel techniques. The extract is dried with sodium sulfate and concentrated.

5.1.1 Gel permeation chromatography (GPC) cleanup is required for PCB or pesticide analysis unless the sample matrix is expected to be clean. After GPC, the sample is concentrated and further cleaned with florisil.

5.1.2 For base neutral acids (BNAs), base neutrals (BNs), acids or polynuclear aromatic hydrocarbons (PNAs), the sample extract is diluted to 1.0 mL in methylene chloride. No further cleanup is required.

5.2 Extracts are then analyzed by gas chromatography (GC) or GC/mass spectrometry (MS) for the requested parameters. See DEQ Laboratory SOP# 500 and SOP #502.

6.0 Definitions

6.1 MDL – The minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.

6.2 Surrogate analyte (SA) - A pure analyte which is extremely unlikely to be found in any sample and which is added to a sample aliquot in known amounts before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method performance with each sample.

6.3 MPB - An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and surrogates that are used with samples.

6.4 Laboratory control sample (LCS) - An aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LCS is treated exactly as a sample including extraction and analysis.

6.5 Lab control sample duplicate (LCSD) – A second aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LCSD is treated exactly as a sample including extraction and analysis.

6.6 Matrix spike (MS) - An aliquot of a sample to which known quantities of the method analytes are added in the laboratory. The MS is treated exactly as a sample including extraction and analysis. The background concentrations of the analytes in the sample must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

6.7 Matrix spike duplicate (MSD) - A second aliquot of sample to which known quantities of the target analytes are added in the laboratory. The MSD is treated exactly as a sample including extraction and analysis. The background concentrations of the analytes in the sample must be determined in a separate aliquot and the measured values in the MSD corrected for background concentrations. Analyses of the MS and MSD indicate precision associated with laboratory procedures.

7.0 Interferences

7.1 Interferences may be from solvents, reagents, glassware, and other sample processing equipment and may cause misinterpretation of GCs and/or elevated reporting limits. All of these materials must be demonstrated to be virtually free from interferences under the conditions of the analysis by running method preparation blanks. The use of pesticide grade or better reagents will reduce the occurrence of interferences.

7.2 Interferences coextracted from samples will vary considerably from source to source depending upon the diversity of the samples. While general cleanup techniques are referenced in this method, unique samples may require additional cleanup approaches.

7.3 Plastics must be avoided due to phthalate ester contamination.

8.0 Safety

8.1 Safety glasses are required in all designated laboratory areas.

8.2 Be familiar with the Laboratory Chemical Hygiene Plan.

8.3 Be familiar with the Laboratory Safety Policy (SOP 100).

8.4 Gas tanks must be secured to a wall or counter top.

8.5 MeCl, hexane, sulfuric acid, methanol, acetone, sodium hydroxide, and sodium sulfate material safety data sheets (MSDSs) are attached.

8.6 Protective gloves must be worn when handling solvents.

8.7 Samples containing cyanide need special care if acid is added to them. Because acid releases cyanide, it must be added in a hood with the sash pulled as far down as possible. Shake the sample and acid. Vent it in a hood. Cyanide smells similar to almonds or bread baking.

8.8 Solvent spill kits are located near the refrigerator in Room 301.

9.0 Equipment and Supplies

9.1 Extraction equipment

9.1.1 Separatory funnel – 2000 mL with teflon (Nalgene part #4301-2000 or equivalent).
9.1.2 FEP teflon filling funnel, 450mL – Big Science, Inc. part #P00007
9.1.3 Glass wool – Pyrex brand or equivalent.
9.1.4 Turbovap tubes – Zymark, 250 mL, 50 mL and suitable racks.
9.1.5 Turbovap workstation – Zymark, model ZW640-3.
9.1.6 Rotomixer – Associated Design and Manufacturing Co.
9.1.7 pH paper – EM-Reagents, colorfast, pH 0-14, or equivalent.
9.1.8 Crimp cap vials and caps – 2 mL.

9.1.8.1 Fill each vial with one mL of MeCl from a calibrated bottle top dispenser. Check the dispenser prior to use with a volumetric flask.
9.1.8.2 Mark the meniscus on each vial.
9.1.8.3 Discard the solvent. Turn the vial upside down in a rack to dry.

9.1.9 Pipettes – Pasteur.
9.1.10 Volumetric pipets – Class A, various sizes.

9.1.11 Volumetric flasks – Class A, various sizes with ground glass stoppers.
9.1.12 Syringes – 50 µl or 100 µl.
9.1.13 Stir bars and stir plate.
9.1.14 Beakers – 1 L.
9.1.15 Graduated cylinders – 2000 mL and 500 mL.
9.1.16 Oven – Capable of maintaining 130°C.
9.1.17 Bottle top dispensers – 10 mL and 50 mL, solvent resistant.
9.1.18 Bottles – Amber glass, Teflon lined screw-cap, glass, 250 mL and 1000 mL.
9.1.19 Centrifuge – Dupont Sorvall GLC-2B or equivalent.
9.1.20 Centrifuge tubes – Glass, must fit into Section 9.1.19.
9.1.21 Pipet bulbs – For pasteur and volumetric pipets.
9.1.22 Tweezers – Long.
9.1.23 Flasks – Erlenmeyer, glass, 250 mL.
9.1.24 Foil.
9.1.25 Culture tubes – VWR #60825-618 or equivalent, glass, 16 x 100 mm.

9.1.25.1 Fill each tube with 10 mL of MeCl or hexane from a calibrated bottle top dispenser. Check the dispenser prior to use with a volumetric flask.
9.1.25.2 Mark the meniscus on each tube.
9.1.25.3 Discard the solvent. Turn the tubes upside down in a rack to dry.

9.1.26 Caps – Plug tite, large, VWR part #15070-176, 1000 per pack.
9.1.27 Nitrile gloves – various sizes.

10.0 Reagents and Standards

10.1 All reagents, solvents, and standards must be traceable to the stock inventory tracking log.

10.2 All reagents, solvents, and standards must be labeled with: expiration date (if applicable) and tracking number.

10.3 All prepared reagents and standards must be labeled with: date prepared, expiration date, preparer's initials, tracking number, diluent, and description.

10.4 All standard logbooks must be completely filled out.

10.5 All certificates of analysis must include the stock inventory tracking number that was assigned to the standard and given to the Quality Control (QC) Officer.

10.6 Sodium sulfate (ACS) – Granular, anhydrous, stored in glass containers at 130°C for a minimum of 12 hours prior to use.

10.7 Solvents – Pesticide residue analysis grade, n-hexane, acetone, technical grade acetone, MeCl, and methanol.

10.8 Reagent water – Organic free.

10.9 Nitrogen – High purity.

10.10 Sulfuric acid (1:1 V/V) H_2SO_4 – Measure 250 mL of reagent water into a 1 L beaker. Add a stir bar. While stirring, slowly add 250 mL of concentrated sulfuric acid. Cool. Pour into a glass reagent bottle. Label. Complete the SE standard logbook.

NOTE: *Never add water to acid.*

10.11 Sodium hydroxide (10 N) NaOH – Measure 400 mL of reagent water into a 1 L beaker. Add a stir bar. While stirring, slowly add 200 g of sodium hydroxide pellets. Cool. Pour into a 500 mL volumetric bottle and dilute to the mark. Mix Well. Label. Complete the SE standard logbook.

NOTE: *Purchased 50% (W/W) NaOH solution may be substituted.*

10.12 Stock standards (SSs) – All standards must be certified reference materials. Manufacturer's certificates of analysis are retained by the DEQ Laboratory and kept as part of the overall quality assurance QA/QC requirements. All SSs must be given a unique lab tracking number and logged into the SS tracking logbook. Store SSs in amber sealed ampules at 10°C to 20°C. Protect the standards from light.

10.13 Surrogate solutions

10.13.1 BN surrogate - Absolute Standards #24016 or equivalent, 5000 ug/mL in MeCl.
2-Fluorobiphenyl
Nitrobenzene-d5
p-Terphenyl-d14

10.13.2 Acid surrogate - Absolute Standards #24015 or equivalent, 10,000 ug/mL in methanol.
2-Fluorophenol
Phenol-d6
2,4,6-Tribromphenol

10.13.3 BNA surrogate solution (BN = 25 ppm, A = 50 ppm)

10.13.3.1 Partially fill a 1000 mL volumetric flask with methanol.
10.13.3.2 Pipette 5 mL of BN surrogate (Section 10.13.1) into the flask.
10.13.3.3 Pipette 5 mL of acid surrogate (Section 10.13.2) into the flask. Mix.
10.13.3.4 Dilute the flask to the mark with methanol. Mix.
10.13.3.5 Transfer to amber screw cap bottles. Label.
10.13.3.6 Store current bottle of solution in a refrigerator and store extra bottles of solution in a freezer. Replace every six months or sooner as needed.

10.13.4 Pesticide/PCB surrogate solution (200 ug/L)

10.13.4.1 Absolute Standards #20023 or equivalent, 200 ppm in acetone.
Decachlorobiphenyl
2,4,5,6-Tetrachloro-m-xylene

10.13.4.2 Partially fill a 1000 mL volumetric flask with methanol.

10.13.4.3 Pipet 1 mL of stock surrogate solution (Section 10.13.4.1) directly into the methanol in the flask. Mix.

10.13.4.4 Dilute the flask to the mark with methanol. Mix.

10.13.4.5 Transfer to amber screw cap bottles. Label.

10.13.4.6 Store current bottle of solution in a refrigerator and store extra bottles of solution in a freezer. Replace every six months or sooner as needed.

10.14 Spike Solutions

10.14.1 BNA spike solution (BN = 50 ug/mL; A = 100 ug/mL)

10.14.1.1 Absolute Standards part #93009, Method 625 with 54 components. Solution is purchased complete and ready to use. See attachment #22.11 for complete compound list.

10.14.1.2 Store current bottle of solution in a refrigerator and store extra bottles of solution in a freezer. Replace every six months or sooner as needed.

10.14.2 Pesticide spike solution

10.14.2.1 Accustandard #S-7179 or equivalent, 10,000 mg/L in methanol.

Aldrin	Endosulfan I
a-BHC	Endosulfan II
b-BHC	Endosulfan sulfate
d-BHC	Endrin
g-BHC (lindane)	Endrin aldehyde
BP-6 (PPB)	Endrine ketone
a-Chlordane	Heptachlor
g-Chlrodane	Heptaclor epoxide
4,4'-DDD	Hexabromobenzene
4,4'-DDE	Methoxyclor
4,4'-DDT	Mirex
Dieldrin	

10.14.2.2 Pesticide spike solution for water samples, 200 ug/L

10.14.2.2.1 Partially fill a 100 mL volumetric flask with methanol.

10.14.2.2.2 Pipet 20 mL from sediment pesticide spike into volumetric (Section 10.14.2.3). Mix.

10.14.2.2.3 Dilute to the mark with methanol. Mix.
10.14.2.2.4 Transfer to a 100 mL amber screw cap bottle. Label.
10.14.2.2.5 Store in a refrigerator. Replace every six months or sooner as needed.

10.14.2.3 Pesticide spike solution for soil samples, 10,000ug/L

10.14.2.3.1 Partially fill a 200 mL volumetric flask with methanol.
10.14.2.3.2 Inject 200 uL of stock pesticide (Section 10.14.2.1) directly into the methanol in the flask.
10.14.2.3.3 Dilute to the mark with methanol. Mix.
10.14.2.3.4 Transfer to a 250 mL amber screw cap bottle. Label.
10.14.2.3.5 Store in a refrigerator. Replace every six months or sooner as needed.

10.14.3 PCB spike solution (Aroclors 1016 and 1260)

10.14.3.1 Aroclor 1016 – Absolute Standards #70015, 1000 ppm in methanol.
10.14.3.2 Aroclor 1260 – Absolute Standards #70021, 1000 ppm in methanol.
10.14.3.3 PCB spike solution (Aroclor 1016, 1ppm and Aroclor 1260, 1 ppm)

10.14.3.3.1 Partially fill a 100 mL volumetric flask with methanol.
10.14.3.3.2 Inject 100 µL of PCB spike (Section 10.14.3.1) directly into the methanol in the flask.
10.14.3.3.3 Inject 100 µL of PCB spike (Section 10.14.3.2) directly into the methanol in the flask. Mix.
10.14.3.3.4 Dilute to the mark with methanol. Mix.
10.14.3.3.5 Transfer to a 100 mL amber screw cap bottle. Label.
10.14.3.3.6 Store solution in a refrigerator. Replace every six months or sooner as needed.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Samples must be collected in 1 L amber glass bottles with teflon or foil lined caps.

11.2 Samples should be stored in ice until the laboratory receives them. They are then refrigerated at 4°C until extraction.

11.3 The U.S. Environmental Protection Agency maximum holding time is seven days from sample collection to extraction.

11.4 Sample extracts should be stored in a freezer and must be analyzed within 40 days of extraction.

12.0 QC

12.1 Sample batches consist of a maximum of 20 samples, 1 method preparation blank, 1 MS, 1 MSD, and 1 LCS. If MS/MSD are not available, an LCSD is also included in the batch.

12.2 A batch may stay open for up to one week, with a maximum or 20 samples. Any additional samples must be accompanied by one MPB, per day extracted. Additional MPBs are not included in the 20 samples.

12.3 MSs and MSDs are actual samples that are selected at random from within the batch of 20 unless requested by field staff.

12.4 All 20 samples and the QC samples are spiked with known surrogate concentrations. The recoveries for these compounds are used as indicators of possible problems that may occur during extraction and cleanup of individual samples.

12.5 The MSs, MSDs, LCSs, and LCSDs are spiked at a known concentration with the analyte compounds. The replicate spikes are used to evaluate the entire batch for precision and accuracy.

12.6 Initial demonstration of ability – Each analyst must demonstrate initial proficiency with this method by generating data of acceptable accuracy and precision for target analytes.

12.6.1 Prepare five sets of glassware for extraction.
12.6.2 Add 1 L of DI water into each of the five separatory funnels.
12.6.3 Adjust pH if applicable and add 1 mL of BNA surrogate (Section 10.13.3) or 1 mL of pesticide/PCB surrogate (Section 10.13.4) to each separatory funnel depending upon the analytes to be extracted.
12.6.4 Spike four of the separatory funnels with 1 mL of BNA spike (see SVOC chemist to obtain a full list spike), or 1 mL of pesticide spike (Section 10.14.2.2) or 1 mL of PCB spike (Section 10.14.3.3) depending upon the analytes to be extracted. Label these as LCS1, LCS2, LCS3, and LCS4.
12.6.5 Label the remaining separatory funnel as MPB.
12.6.6 Extract as in Section 14.2 or Section 14.5 as appropriate.
12.6.7 Follow all cleanup procedures used for routine samples.
12.6.8 Analyze, calculate, and evaluate results by the same procedures used to analyze samples (SOP 500 or SOP 502).

12.7 MDLs

12.7.1 Prepare MDLs at levels specified by the analyst and with the spike solutions they provide.
12.7.2 Prepare nine sets of glassware for extraction.
12.7.3 Add 1 L of DI water to each separatory funnel.

12.7.4 Adjust pH if applicable and add 1 mL of BNA surrogate (Section 10.13.3) or 1 mL of pesticide/PCB surrogate (Section 10.13.4) to each separatory funnel depending upon the analytes to be extracted.

12.7.5 Spike eight of the separatory funnels with 1 mL of the spike provided by the analyst. Label the 8 separatory funnels as MDL1, MDL2, MDL3, MDL4, MDL5, MDL6, MDL7, and MDL8. Label the remaining separatory funnel as MPB.

12.7.6 Extract as in Section 14.2 or Section 14.5 as appropriate.

12.7.7 Follow all cleanup procedures used for routine samples.

12.7.8 Analyze and evaluate results by the same procedures used to analyze samples (SOP 500 or SOP 502).

13.0 Calibration and Standardization

13.1 This section is not applicable to this method.

14.0 Procedure

14.1 Glassware cleaning

14.1.1 Dishwasher

14.1.1.1 Rinse separatory funnels with warm tap water. Wash separatory funnels in sets of four or six.

14.1.1.2 Close the stopcock. Add approximately 100 mL of tap water to one separatory funnel.

14.1.1.3 Add several mLs of concentrated sulfuric acid (H_2SO_4) to the separatory funnel with the water. Swirl to rinse entire funnel. Drain solution through the stopcock into the next separatory funnel. Repeat. Rinse with tap water after acid rinse.

14.1.1.4 Rinse other glassware with H_2SO_4 if necessary.

14.1.1.5 Remove the stopcocks for hand washing. Caps are run through the dishwasher.

14.1.1.6 Load separatory funnels onto the top rack of the dishwasher. They may also be placed on the bottom rack if there is room.

14.1.1.7 Other glassware goes on the bottom rack and must be secured with a rack or screen.

14.1.2 Hand washing

14.1.2.1 Remove felt tip marks from glassware with acetone on a paper towel.

14.1.2.2 Rinse with warm tap water.

14.1.2.3 Wash with hot soapy water (Liqui-Nox dish soap).

14.1.2.4 Rinse with hot tap water.

14.1.2.5 Rinse separatory funnels with H_2SO_4 as in Section 14.1.1.3 through Section 14.1.1.4.

14.1.2.6 Rinse with tap water several times.

14.1.2.7 Remove the stopcocks.

14.1.2.8 Rinse with acetone (technical grade from five gallon can).

14.1.2.9 Drain dry.

14.1.3 Solvent rinsing

14.1.3.1 Rinse all glassware with MeCl or hexane prior to use.

14.2 Organic neutral (pesticides, PCBs, or Scan 3)

14.2.1 Fill in the extraction logbook.
14.2.2 Label separatory funnels and turbovap tubes with the sample ID or QC ID.
14.2.3 Close stopcocks on separatory funnels.
14.2.4 Pour 1 L of deionized (DI) water into each of the separatory funnels labeled MPB, LCS, and LCSD (if applicable).

NOTE: *All samples, surrogates, and spikes must be at room temperature prior to use.*

14.2.5 Pour each sample into the appropriate separatory funnel.

NOTE: *Make sure the meniscus has been marked on each sample bottle.*

14.2.6 Check to make sure that the pH of the samples is between five and seven. Add acid (Section 10.10) or base (Section 10.11) to adjust the pH to five to seven if necessary. Record in logbook.
14.2.7 Add 1 mL (by class A pipette) of pesticide/PCB surrogate (Section 10.13.4) to every separatory funnel.

NOTE: *Make sure it is surrogate that is added.*

14.2.8 Add 1 mL (by class A pipette) of pesticide (Section 10.14.2.2) or PCB spike solution (Section 10.14.3.3) to the LCS, LCSD, MS, and MSD. The choice of spike depends on the parameters being tested. Spike with pesticide spike if pesticides or Scan 3 are requested. Otherwise, spike with PCBs.
14.2.9 Add 60 mL of MeCl to the MPB, LCS, and LCSD separatory funnels.
14.2.10 Add 60 mL of MeCl to each sample bottle. Replace top. Swirl, being careful not to let pressure build up. Pour the MeCl into the respective separatory funnel.
14.2.11 Place caps on each separatory funnel. Remove from stand. Invert. Release pressure into a hood. Shake once. Vent. Shake. Vent until pressure is minimized. Place on rotary extractor. Bungee it down. When extractor is full, rotate several turns. Vent. Turn on extractor. Set timer for five minutes.

Note: *Watch for leaking from separatory funnels. Change caps or stopcocks if necessary. Make a note in the extraction logbook.*

14.2.12 After five minutes have passed, vent each separatory funnel. Replace in stand. Remove tops. Let sit for 15 minutes.
14.2.13 Place glass wool in bottom of each drying funnel. Pour approximately two inches of cooled sodium sulfate into the funnels. Rinse each funnel with MeCl until the solvent runs through. When drained, place a funnel over each turbovap tube.

14.2.14 Drain MeCl layer from separatory funnel into the respective drying funnel. See emulsion section (Section 14.6) if an emulsion is present.

14.2.15 Extract each separatory funnel two more times with 60 mL of MeCl each time. Rinse sample bottles with MeCl as before. Vent. Rotate as before. These last two extractions only need to sit for five minutes each.

14.2.16 Full DEQ amber bottles have an initial volume of 1000 mL. All other sample bottles will need to be filled with tap water to the marked level and then poured into a graduated cylinder for measurement. Record initial volume in the extraction logbook. Rinse all sample bottles with water. Place in boxes for recycling of amber glass.

14.2.17 Rinse each drying funnel with 30 mL of MeCl after the sample has completed draining.

14.2.18 Place foil over each turbovap tube. Use a rubber band to secure.

14.2.19 Place each tube on the turbovap. Check that nitrogen flow is turned on at the wall inlet and minimized at the turbovap. Poke a hole in the foil on each tube. Check that the temperature for the turbovap water bath is set at 35ºC.

14.2.20 Turn on each turbovap. Slowly turn on the nitrogen until samples are swirling. Turn up the nitrogen flow as the sample volume decreases. The nitrogen flow rate should not be allowed to exceed 15/1.0 (seen in the Condition display).

14.2.21 Remove the tubes when the turbovap beeps.

14.2.22 If the samples are not going to be cleaned up by GPC, rinse the walls of the turbovap with 10 mL of hexane (for solvent exchange). Replace the samples on the turbovap. Remove the samples when the turbovap beeps. Transfer them to 1 mL marked vials (Section 9.1.8) with hexane using a pasteur pipet. Place crimp caps on the vials. Label the vial rack with the batch number. Proceed to Section 14.2.24. Place the tray in the freezer located in the pesticide/PCB area.

14.2.23 If the samples are going to be cleaned up by GPC, transfer the turbovap tube contents to a premarked culture tube (Section 9.1.25) using a pasteur pipette. Add MeCl to the turbovap tube. Squirt and rinse the walls of the tube. Transfer to the culture tube, being careful not to go over the 10 mL mark. Dilute to the 10 mL mark. Place a cap (Section 9.1.26) on the culture tube. Remove the label from the turbovap tube. Place on the culture tube. Label the tube rack with the batch number. Place in the refrigerator for GPC cleanup. Proceed to Section 14.2.24, then place the goldenrod sheet in the tray by the GPC computer.

14.2.24 Complete the extraction logbook for the batch. Include all information pertaining to lab accidents, spillage, missed holding times, emulsions, etc. Make a copy on goldenrod paper.

14.3 Acid extractables (phenols)

14.3.1 Fill in the extraction logbook.

14.3.2 Label separatory funnels and turbovap tubes with the sample ID or QC ID.

14.3.3 Close stopcocks on separatory funnels.

14.3.4 Pour 1 L of DI water into each of the separatory funnels labeled MPB, LCS, and LCSD (if applicable).

NOTE: *All samples, surrogates, and spikes must be at room temperature prior to use.*

14.3.5 Pour each sample into the appropriate separatory funnel.

NOTE: *Make sure the volume level has been marked on each sample bottle.*

14.3.6 Add 1 mL of 1:1 H_2SO_4 (Section 10.10) to each separatory funnel. Swirl to mix while keeping the separatory funnel in the hood. Check the pH of each separatory funnel with pH paper to ensure a pH<2. Add more acid if necessary. Note this in the logbook.

14.3.7 Add 1 mL (by class A pipette) of BNA surrogate (Section 10.13.3) to every separatory funnel. Make sure it is surrogate that is added.

14.3.8 Add 1 mL (by class A pipette) of BNA spike solution (Section 10.14.1) to the LCS, LCSD, MS, and MSD.

14.3.9 Add 60 mL of MeCl to the MPB, LCS, and LCSD separatory funnels.

14.3.10 Add 60 mL of MeCl to each sample bottle. Replace top. Swirl, being careful not to let pressure build up. Pour the MeCl into the respective separatory funnel.

14.3.11 Place caps on each separatory funnel. Remove from stand. Invert. Release pressure into a hood. Shake once. Vent. Shake. Vent until pressure is minimized. Place on rotary extractor. Bungee it down. When extractor is full, rotate several turns. Vent. Turn on extractor. Set timer for five minutes.

Note: *Watch for leaking from separatory funnels. Change caps or stopcocks if necessary. Make a note in the extraction logbook.*

14.3.12 After five minutes have passed, vent each separatory funnel. Replace in stand. Remove caps. Let sit for 15 minutes.

14.3.13 Place glass wool in bottom of each drying funnel. Pour approximately two inches of cooled sodium sulfate into the funnels. Rinse each funnel with MeCl until the solvent runs through. When drained, place a funnel over each turbovap tube.

14.3.14 Drain MeCl layer from separatory funnel into the respective drying funnel. See emulsion section (Section 14.6) if an emulsion is present.

14.3.15 Extract each separatory funnel two more times with 60 mL of MeCl each time. Rinse sample bottles with MeCl as before. Vent. Rotate as before. These last two extractions only need to sit for five minutes each.

14.3.16 Label crimp cap glass vials (Section 9.1.8) with sample ID.

14.3.17 Full DEQ amber bottles have an initial volume of 1000 mL. All other sample bottles will need to be filled with tap water to the marked level and then poured into a graduated cylinder for measurement. Record initial volume in the extraction logbook. Rinse all sample bottles with water. Place in boxes for recycling of amber glass.

14.3.18 Rinse each drying funnel with 30 mL of MeCl after the sample has completed draining.

14.3.19 Place foil over each turbovap tube. Use a rubber band to secure.

14.3.20 Place each tube on the turbovap. Check that nitrogen flow is turned on at the wall inlet and minimized at the turbovap. Poke a hole in the foil on each tube. Check that the temperature for the turbovap water bath is set at 35ºC.

14.3.21 Turn on each turbovap. Slowly turn on the nitrogen until samples are swirling. Turn up the nitrogen flow as the sample volume decreases. The nitrogen flow rate should not be allowed to exceed 15/1.0 (seen in the Condition display).

14.3.22 Remove the tubes when the turbovap beeps.

14.3.23 Transfer the turbovap tube contents to the appropriately labeled vial (Section 14.3.16) using a pasteur pipette. Add a small amount (1/2 mL) of MeCl to the turbovap tube. Squirt and rinse the walls of the tube. Transfer to the vial, being careful not to go over the 1 mL mark. Dilute to the 1 mL mark. Place a crimp cap on the vial and crimp.

14.3.24 Complete the extraction logbook for the batch. Include all information pertaining to lab accidents, spillage, missed holding times, emulsions, etc. Make a copy on blue paper.

14.3.25 Label the vial rack with the batch number. Place in the semi-volatile sample freezer. Place the blue sheet in the bin to the right of the other freezer.

14.4 Base neutral-extractables or PNAs only

14.4.1 Fill in the extraction logbook.

14.4.2 Label separatory funnels and turbovap tubes with the sample ID or QC ID.

14.4.3 Close stopcocks on separatory funnels.

14.4.4 Pour 1 L of DI water into each of the separatory funnels labeled MPB, LCS, and LCSD (if applicable).

NOTE: *All samples, surrogates, and spikes must be at room temperature prior to use.*

14.4.5 Pour each sample into the appropriate separatory funnel.

NOTE: *Make sure the volume level has been marked on each sample bottle.*

14.4.6 Add 1 mL of 1:1 H_2SO_4 (Section 10.10) to each separatory funnel. Swirl to mix. Check the pH of each separatory funnel with pH paper to ensure a pH<2. Add more acid if necessary. Note this in the logbook.

14.4.7 Add 1 mL (by class A pipette) of BNA surrogate (Section 10.13.3) to every separatory funnel. Make sure it is surrogate that is added.

14.4.8 Add 1 mL (by class A pipette) of BNA spike solution (Section 10.14.1) to the LCS, LCSD, MS, and MSD.

14.4.9 Add 60 mL of MeCl to the MPB, LCS, and LCSD separatory funnels.

14.4.10 Add 60 mL of MeCl to each sample bottle. Replace top. Swirl, being careful not to let pressure build up. Pour the MeCl into the respective separatory funnel.

14.4.11 Place caps on each separatory funnel. Remove from stand. Invert. Release pressure into a hood. Shake once. Vent. Shake. Vent until pressure is minimized. Place on rotary extractor. Bungee it down. When extractor is full, rotate several turns. Vent. Turn on extractor. Set timer for five minutes.

Note: *Watch for leaking from separatory funnels. Change caps or stopcocks if necessary. Make a note in the extraction logbook.*

14.4.12 After five minutes have passed, vent each separatory funnel. Replace in stand. Remove tops. Let sit for 15 minutes.

14.4.13 Place glass wool in bottom of each drying funnel. Pour approximately two inches of cooled sodium sulfate into the funnels. Rinse each funnel with MeCl until solvent runs through. When drained, place a funnel over each turbovap tube.

14.4.14 Drain MeCl layer from separatory funnel into the respective funnel. See emulsion section (Section 14.6) if an emulsion is present.

14.4.15 Extract each separatory funnel one more time with 60 mL of MeCl. Rinse sample bottles with MeCl as before. Vent. Rotate as before. The second extraction only needs to sit for five minutes. Drain samples.

14.4.16 Add two to three mLs of 10 N NaOH (Section 10.11) to each separatory funnel. Swirl to mix. Check the pH of each separatory funnel with pH paper to ensure a pH>11. Add more base if necessary. Note this in the logbook.

14.4.17 Proceed with third extraction. Repeat steps 14.4.9 through 14.4.12. After the samples have been rotated, allow to sit for 15 minutes before draining extract.

14.4.18 Label crimp cap glass vials (Section 9.1.8) with sample ID.

14.4.19 Full DEQ amber bottles have an initial volume of 1000 mL. All other sample bottles will need to be filled with tap water to the marked level and then poured into a graduated cylinder for measurement. Record initial volume in the extraction logbook. Rinse all sample bottles with water. Place in boxes for recycling of amber glass.

14.4.20 Rinse each drying funnel with 30 mL of MeCl after the sample has completed draining.

14.4.21 Place foil over each turbovap tube. Use a rubber band to secure.

14.4.22 Place each tube on the turbovap. Check that nitrogen flow is turned on at the wall inlet and minimized at the turbovap. Poke a hole in the foil on each tube. Check that the temperature for the turbovap water bath is set at 35ºC.

14.4.23 Turn on each turbovap. Slowly turn on the nitrogen until samples are swirling. Turn up the nitrogen flow as the sample volume decreases. The nitrogen flow rate should not be allowed to exceed 15/1.0 (seen in the Condition display).

14.4.24 Remove the tubes when the turbovap beeps.

14.4.25 Transfer the turbovap tube contents to an appropriately labeled vial (Section 14.4.18) using a pasteur pipette. Add a small amount (1/2 mL) of MeCl to the turbovap tube. Squirt and rinse the walls of the tube. Transfer to the vial, being careful not to go over the 1 mL mark. Dilute to the 1 mL mark. Place a crimp cap on the vial and crimp.

14.4.26 Complete the extraction logbook for the batch. Include all information pertaining to lab accidents, spillage, missed holding times, emulsions, etc. Make a copy on blue paper.

14.4.27 Label the vial rack with the batch number. Place in the semi-volatile sample freezer. Place the blue sheet in the bin to the right of the other freezer.

14.5 BNA extractables

14.5.1 Follow the acid extractables procedure.(Section 14.3) After the third MeCl drain (Section 14.3.15), add two to three mLs of NaOH (Section 10.11) to the separatory funnel. Remove turbovap tubes containing the acid extract and place on the turbovap to concentrate. Proceed as in the BN procedure (Section 14.4), except in the BNA procedure the last 3 extractions will all be basic.

14.5.2 Collect the BN fraction in a 250 mL rinsed and labeled erlenmeyer flask.

14.5.3 Add the BN fraction to the acid fraction in the turbovap tubes when the acid fraction volume is low.

14.5.4 Evaporate the combined extract as in the acid or BN sections.

14.6 Emulsions

14.6.1 An emulsion occurs when there is not complete separation of the MeCl layer from the water layer.

14.6.2 Try swirling the separatory funnel to break up the emulsion.

14.6.3 Draw off the MeCl layer into a centrifuge tube. Sometimes this action will break the emulsion up, and the MeCl layer can be transferred to the drying funnel with a disposable pipette, and the water can be returned to the separatory funnel.

14.6.4 If the emulsion still persists, centrifuge for five minutes at approximately 3000 rev/minute. Remove the MeCl layer with a disposable pipette. Return the water layer to the separatory funnel.

14.6.5 If the emulsion still persists, add extra MeCl to the tube. Shake. Vent. Make sure the cap does not leak. Centrifuge again. This information must be recorded in the extraction logbook. Remove the MeCl layer to the drying funnel. Return the water layer to the separatory funnel.

14.6.6 For more difficult emulsions, sodium sulfate can be added to the centrifuge tube to separate the water from the MeCl layer.

14.7 Low level PCB extraction

14.7.1 Three 1 L amber glass bottles are required per sample for low level PCB extraction.

14.7.2 Fill in the extraction logbook.

14.7.3 Label separatory funnels and turbovap tubes with the QC ID. Label two separatory funnels and two turbovap tubes per sample with the sample number. Add an “A” to one and add a “B” to the other.

14.7.4 Close stopcocks on separatory funnels.

14.7.5 Pour 1 L of DI water into each of the separatory funnels labeled MPB, LCS, and LCSD (if applicable).

NOTE: *All samples, surrogates, and spikes must be at room temperature prior to use.*

14.7.6 Divide the three ambers of sample between the two labeled separatory funnels.

NOTE: *Make sure the meniscus has been marked on each sample bottle.*

14.7.7 Check that the pH of all the samples is between five and seven. Add acid (Section 10.10) or base (Section 10.11) to adjust the pH to five to seven if necessary. Note in the extraction logbook.

14.7.8 Add 1 mL (by class A pipette) of pesticide/PCB surrogate (Section 10.13.4) to the QC separatory funnels. (MPB, LCS, LCSD, MS, MSD) Add ½ mL of surrogate (Section 10.13.4) to each sample separatory funnel.

NOTE: *Make sure it is surrogate that is added.*

14.7.9 Add 1 mL (by class A pipette) of PCB spike solution (Section 10.14.3.3) to the LCS, LCSD, MS, and MSD.

14.7.10 Add 60 mL of MeCl to the MPB, LCS, and LCSD separatory funnels.

14.7.11 Add 120 mL of MeCl to each sample bottle. Replace tops. Swirl, being careful not to let pressure build up. Use the MeCl of one amber per rotation and divide between the two separatory funnels, giving each of the three ambers one rinse.

14.7.12 Place caps on each separatory funnel. Remove from stand. Invert. Release pressure into a hood. Shake once. Vent. Shake. Vent until pressure is minimized. Place on rotary extractor. Bungee it down. When extractor is full, rotate several turns. Vent. Turn on extractor. Set timer for five minutes.

Note: *Watch for leaking from separatory funnels. Change caps or stopcocks if necessary. Make a note in the extraction logbook.*

14.7.13 After five minutes have passed, vent each separatory funnel. Replace in stand. Remove caps. Let sit for 15 minutes.

14.7.14 Place glass wool in bottom of each drying funnel. Pour approximately two inches of cooled sodium sulfate into the funnels. Rinse each column with MeCl until solvent runs through. When drained, place a funnel over each turbovap tube.

14.7.15 Drain MeCl layer from separatory funnel into the respective funnel. See emulsion section (Section 14.6) if an emulsion is present.

14.7.16 Extract each separatory funnel two more times with 60 mL of MeCl each time. Vent. Rotate as before. These last two extractions only need to sit for five minutes each.

14.7.17 Full DEQ amber bottles have an initial volume of 1000 mL. All other sample bottles will need to be filled with tap water to the marked level and then poured into a graduated cylinder for measurement. Record initial volume in the extraction logbook. Rinse all sample bottles with water. Place in boxes for recycling of amber glass.

14.7.18 Rinse each drying funnel with 30 mL of MeCl after the sample has completed draining.

14.7.19 Place foil over each turbovap tube. Use a rubber band to secure.

14.7.20 Place each tube on the turbovap. Check that nitrogen flow is turned on at the wall inlet and minimized at the turbovap. Poke a hole in the foil on each tube. Check that the temperature for the turbovap water bath is set at 35ºC.

14.7.21 Turn on each turbovap. Slowly turn on the nitrogen until samples are swirling. Turn up the nitrogen flow as the sample volume decreases. The nitrogen flow rate should not be allowed to exceed 15/1.0 (seen in the Condition display).

14.7.22 Remove the tubes when the turbovap beeps.

14.7.23 Combine the two extracts for each sample (Section 14.7.3) into one extract by transferring both extracts into the same premarked culture tube (Section 9.1.25) using a Pasteur pipette. Add MeCl to the turbovap tubes. Squirt and rinse the walls of the tube. Transfer to the culture tube, being careful not to go over the 10 mL mark. Dilute to the 10 mL mark. Place a cap (Section 9.1.26) on the culture tube. Remove the label from the turbovap tube. Place on the culture tube.

14.7.24 Complete the extraction logbook for the batch. Include all information pertaining to lab accidents, spillage, missed holding times, emulsions, etc. Make a copy on goldenrod paper.

14.7.25 Label the tube rack with the batch number. Place in the refrigerator for GPC cleanup. Place the goldenrod sheet in the basket next to the GPC computer.

15.0 Calculations

15.1 This section is not applicable to this method.

16.0 Method Performance

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16.1 This section is not applicable to this method.

17.0 Pollution Prevention

17.1 For information concerning pollution prevention which may be applicable to laboratory operations, consult Less is Better: Laboratory Chemical Management for Waste Reduction available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, DC 20036.

18.0 Data Assessment

18.1 Samples must be extracted within seven days of collection. If samples are extracted past the holding time, it must be noted on the paperwork.

18.2 LIMS entry: Creating a batch.

18.2.1 Select the Labworks CITRIX icon on the desktop.
18.2.2 Enter your User ID and password. Select the ENV PRD database. Click "OK".
18.2.3 Click on "QA Batch".
18.2.4 Select "New Batches", then "Batch by Analysis".
18.2.5 Enter analysis in white box. Either E_WBA or E_PPW. Click "OK".
18.2.6 Select "OK" again. At this point, a list of possible samples for that analysis will appear. The samples included in the batch need to be marked with a red check. Uncheck those samples that are not with the batch.
18.2.7 Select "OK".
18.2.8 Make sure that the batch size matches or is greater than the number of samples. Select "OK".
18.2.9 Change the default batch name to match the extractions batch number by changing the last four numbers only. Example: from default number E_PPW-5111 to actual batch number E_PPW-4577.
18.2.10 Click on the sample that is being used for QC. Change QA Group and QA Tests as needed.
18.2.11 Select "OK".
18.2.12 Select "Exit".

18.3 LIMS entry: Entering results.

18.3.1 Select the Labworks CITRIX icon on the desktop.

18.3.2 Enter your User ID and password. Select the ENV_PRD database. Select "OK".
18.3.3 Select "Results".
18.3.4 Results entry template should read "Extract". Select "QA/QC Batch".
18.3.5 Select appropriate analysis code. Either E_WBA or E_PPW. Click "OK".
18.3.6 Select appropriate batch. Click on "OK" twice.
18.3.7 Enter the samples initial volume under "Results", check that the dates and initials are correct. Make any changes on the first sample; all following samples will be the same. Add any Qualifier Codes to the "Qualify" line. Find the QC sample. Scroll down and enter MS/MSD volumes, if applicable, and surrogate and spike tracking numbers. Select "Save".
18.3.8 Select "Exit".

18.4 Backlog Reports

18.4.1 Select the Labworks CITRIX icon on the desktop.
18.4.2 Enter your User ID and password. Select the ENV_PRD database. Select "OK".
18.4.3 Select "Backlog".
18.4.4 Search backlog by "Department Code". Select "OK".
18.4.5 Enter O_WA into the white bar. Select "OK".
18.4.6 Select "Display Report".
18.4.7 Select "Close" then "Exit" when done.

19.0 Corrective Actions

19.1 If surrogate recoveries fall outside the acceptance limits and the sample is within the seven-day hold time, reextraction is performed if another amber bottle is available.

19.2 All data reported for samples extracted outside the hold time receive a hold time code.

20.0 Waste Management

20.1 The Laboratory Safety Officer arranges for proper waste disposal.

21.0 References

21.1 United States Code of Federal Regulations, Title 40, U.S. Government Printing Office, Washington, D.C., Appendix A & B.

21.2 Test Methods for Evaluating Solid Waste, U.S. Environmental Protection Agency, Document SW-846, Third Edition, 1986, Final Update III, December 1996.

21.3 Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, EPA-600/4-79-020, March 1983.

22.0 Attachments

22.1 MeCl MSDS.

22.2 Hexane MSDS.

22.3 Sulfuric acid MSDS.

22.4 Sodium hydroxide MSDS.

22.5 Sodium sulfate MSDS.

22.6 Methanol MSDS.

22.7 Acetone MSDS.

22.8 Semi-volatile standard logbook.

22.9 Stock chemical logbook.

22.10 Water extraction logbook.

22.11 BNA spike compound list.

MDEQ SOP# 405 REVISION # 2

APPROVED BY _____ DATE 1/12/2005
CHIEF, LABORATORY SERVICES SECTION

APPROVED BY _____ DATE 1/24/05
COMPLIANCE OFFICER

APPROVED BY _____ DATE 12/27/04
QA OFFICER

APPROVED BY _____ DATE 12-20-04
UNIT MANAGER

APPROVED BY _____ DATE 8/27/04
ANALYST

ATTACHMENT 22.1

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MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 202-483-7616

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

METHYLENE CHLORIDE

MSDS Number: M4420 --- *Effective Date: 06/30/98*

1. Product Identification

Synonyms: MC; Dichloromethane (DCM); Methylene dichloride; Methylene bichloride; Methane dichloride
CAS No.: 75-09-2
Molecular Weight: 84.93
Chemical Formula: CH2Cl2
Product Codes: 9264, 9266, 9295, 9315, 9324, 9329, 9330, 9341, 9348, 9350, 9965, Q480

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Methylene Chloride	75-09-2	> 99%	Yes

3. Hazards Identification

Emergency Overview

WARNING! HARMFUL IF SWALLOWED, INHALED OR ABSORBED THROUGH SKIN. AFFECTS CENTRAL NERVOUS SYSTEM, LIVER, CARDIOVASCULAR SYSTEM, AND BLOOD. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. SUSPECT CANCER HAZARD. MAY CAUSE CANCER. Risk of cancer depends on level and duration of exposure.

ATTACHMENT 22.1

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Cancer Causing)
Flammability Rating: 1 - Slight
Reactivity Rating: 1 - Slight
Contact Rating: 2 - Moderate
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: Blue (Health)

Potential Health Effects

Inhalation:
Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes formation of carbon monoxide in blood which affects cardiovascular system and central nervous system. Continued exposure may cause increased light-headedness, staggering, unconsciousness, and even death. Exposure may make the symptoms of angina (chest pains) worse.

Ingestion:
May cause irritation of the gastrointestinal tract with vomiting. If vomiting results in aspiration, chemical pneumonia could follow. Absorption through gastrointestinal tract may produce symptoms of central nervous system depression ranging from light headedness to unconsciousness.

Skin Contact:
Causes irritation, redness and pain. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Eye Contact:
Vapors can cause eye irritation. Contact can produce pain, inflammation and temporal eye damage.

Chronic Exposure:
Can cause headache, mental confusion, depression, liver effects, kidney effects, bronchitis, loss of appetite, nausea, lack of balance, and visual disturbances. Can cause dermatitis upon prolonged skin contact. Methylene chloride may cause cancer in humans.

Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders, eye problems, impaired liver, kidney, respiratory or cardiovascular function may be more susceptible to the effects of this substance.

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.
Ingestion:
If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:
Immediately flush skin with plenty of soap and water for at least 15 minutes while removing contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

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5. Fire Fighting Measures

Fire:
Autoignition temperature: 556C (1033F)
Flammable limits in air % by volume:
lel: 12; uel: 23

Forms flammable vapor-air mixtures above 100C (212F).
Explosion:
Concentrated can be ignited by a high intensity ignition source. Vapor may form flammable mixture in atmosphere that contains a high percentage of oxygen. Sealed containers may rupture when heated.
Fire Extinguishing Media:
Dry chemical, foam or carbon dioxide. Water spray may be used to keep fire exposed containers cool.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Combustion by-products include phosgene and hydrogen chloride gases. Structural firefighters' clothing provides only limited protection to the combustion products of this material.

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6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste

container. Do not use combustible materials, such as saw dust. Do not flush to sewer! US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

7. Handling and Storage

Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect against physical damage. Isolate from any source of heat or ignition. Outside or detached storage is recommended. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product. To minimize decomposition, all storage containers should be galvanized or lined with a phenolic coating. This material may corrode plastic and rubber. Wear special protective equipment (Sec. 8) for maintenance break-in or where exposures may exceed established exposure levels. Wash hands, face, forearms and neck when exiting restricted areas. Shower, dispose of outer clothing, change to clean garments at the end of the day. Avoid cross-contamination of street clothes. Wash hands before eating and do not eat, drink, or smoke in workplace. Odor Threshold: 205 - 307 ppm. The odor threshold only serves as a warning of exposure; not smelling it does not mean you are not being exposed.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
Methylene Chloride (Dichloromethane):
- OSHA Permissible Exposure Limit (PEL) -
25 ppm (TWA), 125 ppm (STEL), 12.5 ppm (8-hour TWA - Action Level)
- ACGIH Threshold Limit Value (TLV) -
50 ppm (TWA), A2 - suspected human carcinogen.

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. The cartridges recommended for this material have a predicted service of less than 30 minutes at concentrations of ten times (10x) the exposure limits. Actual service life will vary considerbly, depending on concentration levels, temperature, humidity, and work rate. This substance has poor warning properties.

Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact. Neoprene is a recommended material

for personal protective equipment. Natural rubber and polyvinyl chloride ARE NOT recommended materials for personal protective equipment.

Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

Other Control Measures:
Do not use closed circuit rebreathing system employing soda lime or other carbon dioxide absorber because of formation of toxic compounds capable of producing cranial nerve paralysis. See OSHA Standard for medical surveillance, record keeping, and reporting requirements for methylene chloride (29 CFR 1910.1052).

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9. Physical and Chemical Properties

Appearance:
Clear, colorless liquid.

Odor:
Chloroform-like odor.

Solubility:
1.32 gm/100 gm water @ 20C.

Specific Gravity:
1.33 @ 15C/4C

pH:
No information found.

% Volatiles by volume @ 21C (70F):
100

Boiling Point:
39.8C (104F)

Melting Point:
-97C (-143F)

Vapor Density (Air=1):
2.9

Vapor Pressure (mm Hg):
350 @ 20C (68F)

Evaporation Rate (BuAc=1):
27.5

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10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage.

Hazardous Decomposition Products:
Emits highly toxic fumes of phosgene when heated to decomposition. Decomposes in a flame or hot surface to form toxic gas phosgene and corrosive mists of hydrochloric acid. Carbon dioxide and carbon monoxide may form when heated to decomposition.

Hazardous Polymerization:
Will not occur.

Incompatibilities:
Strong oxidizers, strong caustics, plastics, rubber, nitric acid, water + heat, and chemically active metals, such as aluminum and magnesium powder, sodium, potassium, and lithium. Avoid contact with open flames and electrical arcs. Liquid methylene chloride will attack some forms of plastics, rubber, and coatings.
Conditions to Avoid:
Moisture, heat, flames, ignition sources and incompatibles.

11. Toxicological Information

Toxicological Data:
Dichloromethane: Oral rat LD50: 1600 mg/kg; inhalation rat LC50: 52 gm/m3; investigated as a tumorigen, mutagen, reproductive effector.
Reproductive Toxicity:
Dichloromethane has been linked to spontaneous abortions in humans.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Methylene Chloride (75-09-2)	No	Yes	2B

12. Ecological Information

Environmental Fate:
When released into the soil, this material may leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material may biodegrade to a moderate extent. When released to water, this material is expected to quickly evaporate. This material has a log octanol-water partition coefficient of less than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material may be moderately degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material is expected to have a half-life of greater than 30 days. When released into the air, this material may be removed from the atmosphere to a moderate extent by wet deposition.
Environmental Toxicity:
The LC50/96-hour values for fish are over 100 mg/l. This material is not expected to be toxic to aquatic life.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: DICHLOROMETHANE
Hazard Class: 6.1
UN/NA: UN1593
Packing Group: III
Information reported for product/size: 52L

International (Water, I.M.O.)

Proper Shipping Name: DICHLOROMETHANE
Hazard Class: 6.1
UN/NA: UN1593
Packing Group: III
Information reported for product/size: 52L

International (Air, I.C.A.O.)

Proper Shipping Name: DICHLOROMETHANE
Hazard Class: 6.1
UN/NA: UN1593
Packing Group: III
Information reported for product/size: 52L

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Methylene Chloride (75-09-2)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

Ingredient	Korea	Canada-- DSL	Canada-- NDSL	Phil.
Methylene Chloride (75-09-2)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\--------

Ingredient	-SARA 302- RQ	-SARA 302- TPQ	-SARA 313- List	-SARA 313- Chemical Catg.
Methylene Chloride (75-09-2)	No	No	Yes	No

--------\Federal, State & International Regulations - Part 2\--------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Methylene Chloride (75-09-2)	1000	U080	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: No Pressure: No
Reactivity: No (Pure / Liquid)

WARNING:
THIS PRODUCT CONTAINS A CHEMICAL(S) KNOWN TO THE STATE OF CALIFORNIA TO CAUSE CANCER.

Australian Hazchem Code: 2Z
Poison Schedule: S5
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

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16. Other Information

NFPA Ratings: Health: **2** Flammability: **1** Reactivity: **0**
Label Hazard Warning:
WARNING! HARMFUL IF SWALLOWED, INHALED OR ABSORBED THROUGH SKIN. AFFECTS CENTRAL NERVOUS SYSTEM, LIVER, CARDIOVASCULAR SYSTEM, AND BLOOD. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. SUSPECT CANCER HAZARD. MAY CAUSE CANCER. Risk of cancer depends on level and duration of exposure.
Label Precautions:
Do not breathe vapor.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Keep away from heat and flame.
Do not get in eyes, on skin, or on clothing.
Label First Aid:
If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. In all cases, get medical attention.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 1, 2.
Disclaimer:
****

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document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.

****

Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

ATTACHMENT 22.2

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MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 202-483-7616

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

HEXANES

MSDS Number: H2379 --- *Effective Date: 04/15/99*

1. Product Identification

Synonyms: Normal Hexane; Hexyl Hydride
CAS No.: 110-54-3 (n-hexane)
Molecular Weight: 86.18
Chemical Formula: CH3(CH2)4CH3 n-hexane
Product Codes: 9277, 9306, 9309, N169

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Hexane	110-54-3	> 95%	Yes
Methylcyclopentane	96-37-7	1 - 2%	Yes
2-Methylpentane	107-83-5	1 - 2%	No
3-Methylpentane	96-14-0	1 - 2%	No
Pentane	109-66-0	0 - 2%	No

3. Hazards Identification

Emergency Overview

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Life)
Flammability Rating: 4 - Extreme (Flammable)
Reactivity Rating: 2 - Moderate
Contact Rating: 2 - Moderate
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

The health hazards addressed are for the major component: n-hexane.

Inhalation:
Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Greater exposure may cause muscle weakness, numbness of the extremities, unconsciousness and death.
Ingestion:
May produce abdominal pain, nausea. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms expected to parallel inhalation.
Skin Contact:
May cause redness, irritation, with dryness, cracking.
Eye Contact:
Vapors may cause irritation. Splashes may cause redness and pain.
Chronic Exposure:
Repeated or prolonged skin contact may defat the skin and produce irritation and dermatitis. Chronic inhalation may cause peripheral nerve disorders and central nervous system effects.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance. May affect the developing fetus.

---

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:
Aspiration hazard. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Get medical attention

immediately.
Skin Contact:
Remove any contaminated clothing. Wipe off excess from skin. Wash skin with soap and water for at least 15 minutes. Get medical attention if irritation develops or persists.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

Note to Physician:
BEI=2,5-hexadione in urine, sample at end of shift at workweeks end, 5 mg/g creatine. Also, measure n-hexane in expired air. Analgesics may be necessary for pain management, there is no specific antidote. Monitor arterial blood gases in cases of severe aspiration.

---

5. Fire Fighting Measures

Fire:
Flash point: -21C (-6F) CC
Autoignition temperature: 224C (435F)
Flammable limits in air % by volume:
lel: 1.1; uel: 7.5
Extremely Flammable Liquid and Vapor! Vapor may cause flash fire. Dangerous fire hazard when exposed to heat or flame.
Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Contact with oxidizing materials may cause extremely violent combustion.Explodes when mixed @ 28C with dinitrogen tetraoxide. Sensitive to static discharge.
Fire Extinguishing Media:
Dry chemical, foam or carbon dioxide. Water may be ineffective.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Water spray may be used to keep fire exposed containers cool. Vapors can flow along surfaces to distant ignition source and flash back. Vapor explosion hazard exists indoors, outdoors, or in sewers.

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6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel

attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB(R) solvent adsorbent is recommended for spills of this product.

---

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from direct sunlight and any area where the fire hazard may be acute. Store in tightly closed containers (preferably under nitrogen atmosphere). Outside or detached storage is preferred. Inside storage should be in a standard flammable liquids storage room or cabinet. Separate from oxidizing materials. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
N-Hexane [110-54-3]:
-OSHA Permissible Exposure Limit (PEL): 500 ppm (TWA)
-ACGIH Threshold Limit Value (TLV): 50 ppm (TWA), Skin
other isomers of hexane
-ACGIH Threshold Limit Value (TLV): 500 ppm (TWA),1000ppm (STEL)
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. This substance has poor warning properties.
Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.
Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

The following properties are for n-hexane, the major component.
Appearance:
Clear, colorless liquid.
Odor:
Gasoline-like odor.
Solubility:
Insoluble in water.
Specific Gravity:
0.66
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100
Boiling Point:
ca. 68C (ca. 154F)
Melting Point:
ca. -95C (ca. -139F)
Vapor Density (Air=1):
3.0
Vapor Pressure (mm Hg):
150 @ 25C (77F)
Evaporation Rate (BuAc=1):
9

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10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Heat will contribute to instability.
Hazardous Decomposition Products:
May produce acrid smoke and irritating fumes when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Strong oxidizers.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

---

11. Toxicological Information

N-Hexane: Oral rat LD50: 28710 mg/kg. Irritation eye rabbit: 10 mg mild. Investigated as a tumorigen, mutagen and reproductive effector.

ATTACHMENT 22.2

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Hexane (110-54-3)	No	No	None
Methylcyclopentane (96-37-7)	No	No	None
2-Methylpentane (107-83-5)	No	No	None
3-Methylpentane (96-14-0)	No	No	None
Pentane (109-66-0)	No	No	None

12. Ecological Information

Environmental Fate:
When released into the soil, this material may biodegrade to a moderate extent. When released into the soil, this material is not expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material may biodegrade to a moderate extent. When released to water, this material is expected to quickly evaporate. When released into the water, this material is expected to have a half-life between 1 and 10 days. This material has an estimated bioconcentration factor (BCF) of less than 100. This material has a log octanol-water partition coefficient of greater than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material is expected to be readily degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material is expected to have a half-life between 1 and 10 days.
Environmental Toxicity:
No information found.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: HEXANES
Hazard Class: 3
UN/NA: UN1208
Packing Group: II
Information reported for product/size: 215L

International (Water, I.M.O.)

Proper Shipping Name: HEXANES
Hazard Class: 3.1
UN/NA: UN1208
Packing Group: II
Information reported for product/size: 215L

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15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Hexane (110-54-3)	Yes	Yes	Yes	Yes
Methylcyclopentane (96-37-7)	Yes	Yes	No	Yes
2-Methylpentane (107-83-5)	Yes	Yes	Yes	Yes
3-Methylpentane (96-14-0)	Yes	Yes	Yes	Yes
Pentane (109-66-0)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Hexane (110-54-3)	Yes	Yes	No	Yes
Methylcyclopentane (96-37-7)	Yes	Yes	No	Yes
2-Methylpentane (107-83-5)	Yes	Yes	No	Yes
3-Methylpentane (96-14-0)	Yes	Yes	No	Yes
Pentane (109-66-0)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\--------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Hexane (110-54-3)	No	No	Yes	No
Methylcyclopentane (96-37-7)	No	No	No	No
2-Methylpentane (107-83-5)	No	No	No	No
3-Methylpentane (96-14-0)	No	No	No	No
Pentane (109-66-0)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\--------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Hexane (110-54-3)	5000	No	No
Methylcyclopentane (96-37-7)	No	No	No
2-Methylpentane (107-83-5)	No	No	No
3-Methylpentane (96-14-0)	No	No	No
Pentane (109-66-0)	No	No	Yes

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: Yes Pressure: No
Reactivity: No (Mixture / Liquid)

Australian Hazchem Code: 3[Y]E
Poison Schedule: No information found.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.
Label Precautions:
Keep away from heat, sparks and flame.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor or mist.
Avoid contact with eyes, skin and clothing.
Label First Aid:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. In all cases call a physician.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 8. Updated the ACGIH part of the Airborne section of TAB 8.
Disclaimer:
****

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****

Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

ATTACHMENT 22.3

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MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

MALLINCKRODT J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 202-483-7616

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

SULFURIC ACID, 52 - 100 %

MSDS Number: S8234 --- *Effective Date: 09/14/00*

1. Product Identification

Synonyms: Oil of vitriol; Babcock acid; sulphuric acid
CAS No.: 7664-93-9
Molecular Weight: 98.08
Chemical Formula: H2SO4 in H2O
Product Codes:
J.T. Baker: 5030, 5137, 5374, 5802, 5815, 5889, 5960, 5961, 5971, 6902, 9673, 9674, 9675, 9676, 9679, 9680, 9681, 9682, 9684, 9687, 9691, 9693, 9694
Mallinckrodt: 2468, 2876, 2878, 2900, 2904, 3780, 4222, 5524, 5557, H644, H976, H996, V344, V651

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Sulfuric Acid	7664-93-9	52 - 100%	Yes
Water	7732-18-5	0 - 48%	No

3. Hazards Identification

Emergency Overview

POISON! DANGER! CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED OR

CONTACTED WITH SKIN. HARMFUL IF INHALED. AFFECTS TEETH. WATER REACTIVE. CANCER HAZARD. STRONG INORGANIC ACID MISTS CONTAINING SULFURIC ACID CAN CAUSE CANCER. Risk of cancer depends on duration and level of exposure.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Poison)
Flammability Rating: 0 - None
Reactivity Rating: 3 - Severe (Water Reactive)
Contact Rating: 4 - Extreme (Corrosive)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: White (Corrosive)

Potential Health Effects

Inhalation:
Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. May cause lung edema, a medical emergency.
Ingestion:
Corrosive. Swallowing can cause severe burns of the mouth, throat, and stomach, leading to death. Can cause sore throat, vomiting, diarrhea. Circulatory collapse with clammy skin, weak and rapid pulse, shallow respirations, and scanty urine may follow ingestion or skin contact. Circulatory shock is often the immediate cause of death.
Skin Contact:
Corrosive. Symptoms of redness, pain, and severe burn can occur. Circulatory collapse with clammy skin, weak and rapid pulse, shallow respirations, and scanty urine may follow skin contact or ingestion. Circulatory shock is often the immediate cause of death.

Eye Contact:
Corrosive. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Chronic Exposure:
Long-term exposure to mist or vapors may cause damage to teeth. Chronic exposure to mists containing sulfuric acid is a cancer hazard.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance.

---

4. First Aid Measures

Inhalation:

Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician immediately.

Ingestion:

DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Call a physician immediately.

Skin Contact:

In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Excess acid on skin can be neutralized with a 2% solution of bicarbonate of soda. Call a physician immediately.

Eye Contact:

Immediately flush eyes with gentle but large stream of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Call a physician immediately.

---

5. Fire Fighting Measures

Fire:

Concentrated material is a strong dehydrating agent. Reacts with organic materials and may cause ignition of finely divided materials on contact.

Explosion:

Contact with most metals causes formation of flammable and explosive hydrogen gas.

Fire Extinguishing Media:

Dry chemical, foam or carbon dioxide. Do not use water on material. However, water spray may be used to keep fire exposed containers cool.

Special Information:

In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Structural firefighter's protective clothing is ineffective for fires involving this material. Stay away from sealed containers.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Neutralize with alkaline material (soda ash, lime), then absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker NEUTRASORB(R) or TEAM(R) 'Low Na+' acid neutralizers are recommended for spills of this product.

---

7. Handling and Storage

Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Protect from physical damage. Keep out of direct sunlight and away from heat, water, and incompatible materials. Do not wash out container and use it for other purposes. When diluting, always add the acid to water; never add water to the acid. When opening metal containers, use non-sparking tools because of the possibility of hydrogen gas being present. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
For Sulfuric Acid:
- OSHA Permissible Exposure Limit (PEL) -
1 mg/m3 (TWA)
- ACGIH Threshold Limit Value (TLV) -
1 mg/m3(TWA), 3 mg/m3 (STEL), A2 - suspected human carcinogen for sulfuric acid contained in strong inorganic acid mists.

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded and engineering controls are not feasible, a full facepiece respirator with an acid gas cartridge and particulate filter (NIOSH type N100 filter) may be worn up to 50 times the exposure limit, or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. If oil particles (e.g. lubricants, cutting fluids, glycerine, etc.) are present, use a NIOSH type R or P particulate filter. For emergencies or instances where the exposure levels are not known, use a full-facepiece positive-pressure, air-supplied respirator. WARNING: Air purifying respirators do not protect workers in oxygen-deficient atmospheres. Where respirators are required, you must have a written program covering the basic requirements in the OSHA respirator standard. These include training, fit testing, medical approval, cleaning, maintenance, cartridge change schedules, etc. See 29CFR1910.134 for details.

Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

Appearance:
Clear oily liquid.
Odor:
Odorless.
Solubility:
Miscible with water, liberates much heat.
Specific Gravity:
1.84 (98%), 1.40 (50%), 1.07 (10%)
pH:
1 N solution (ca. 5% w/w) = 0.3; 0.1 N solution (ca. 0.5% w/w) = 1.2; 0.01 N solution (ca. 0.05% w/w) = 2.1.
% Volatiles by volume @ 21C (70F):
No information found.
Boiling Point:
ca. 290C (ca. 554F) (decomposes at 340C)
Melting Point:
3C (100%), -32C (93%), -38C (78%), -64C (65%).
Vapor Density (Air=1):
3.4
Vapor Pressure (mm Hg):
1 @ 145.8C (295F)
Evaporation Rate (BuAc=1):
No information found.

---

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Concentrated solutions react violently with water, spattering and liberating heat.
Hazardous Decomposition Products:
Toxic fumes of oxides of sulfur when heated to decomposition. Will react with water or steam to produce toxic and corrosive fumes. Reacts with carbonates to generate carbon dioxide gas, and with cyanides and sulfides to form poisonous hydrogen cyanide and hydrogen sulfide respectively.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Water, potassium chlorate, potassium perchlorate, potassium permanganate, sodium, lithium, bases, organic material, halogens, metal acetylides, oxides and hydrides, metals (yields hydrogen gas), strong oxidizing and reducing agents and many other reactive substances.
Conditions to Avoid:
Heat, moisture, incompatibles.

---

11. Toxicological Information

Toxicological Data:
Oral rat LD50: 2140 mg/kg; inhalation rat LC50: 510 mg/m3/2H; standard Draize, eye rabbit, 250 ug (severe); investigated as a tumorigen, mutagen, reproductive effector.
Carcinogenicity:
Cancer Status: The International Agency for Research on Cancer (IARC) has classified "strong inorganic acid mists containing sulfuric acid" as a known human carcinogen, (IARC category 1). This classification applies only to mists containing sulfuric acid and not to sulfuric acid or sulfuric acid solutions.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Sulfuric Acid (7664-93-9)	No	No	None
Water (7732-18-5)	No	No	None

12. Ecological Information

Environmental Fate:
When released into the soil, this material may leach into groundwater. When released into the air, this material may be removed from the atmosphere to a moderate extent by wet deposition. When released into the air, this material may be removed from the atmosphere to a moderate extent by dry deposition.
Environmental Toxicity:
LC50 Flounder 100 to 330 mg/l/48 hr aerated water/Conditions of bioassay not specified; LC50 Shrimp 80 to 90 mg/l/48 hr aerated water /Conditions of bioassay not specified; LC50 Prawn 42.5 ppm/48 hr salt water /Conditions of bioassay not specified. This material may be toxic to aquatic life.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

ATTACHMENT 22.3

Proper Shipping Name: SULFURIC ACID (WITH MORE THAN 51% ACID)
Hazard Class: 8
UN/NA: UN1830
Packing Group: II
Information reported for product/size: 440LB

International (Water, I.M.O.)

Proper Shipping Name: SULPHURIC ACID (WITH MORE THAN 51% ACID)
Hazard Class: 8
UN/NA: UN1830
Packing Group: II
Information reported for product/size: 440LB

---

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Sulfuric Acid (7664-93-9)	Yes	Yes	Yes	Yes
Water (7732-18-5)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Sulfuric Acid (7664-93-9)	Yes	Yes	No	Yes
Water (7732-18-5)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\----------------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Sulfuric Acid (7664-93-9)	1000	1000	Yes	No
Water (7732-18-5)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\----------------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Sulfuric Acid (7664-93-9)	1000	No	No
Water (7732-18-5)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: Yes
SARA 311/312: Acute: Yes Chronic: Yes Fire: No Pressure: No
Reactivity: Yes (Pure / Liquid)

Australian Hazchem Code: 2P
Poison Schedule: No information found.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled

Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **3** Flammability: **0** Reactivity: **2** Other: **Water reactive**
Label Hazard Warning:
POISON! DANGER! CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED OR CONTACTED WITH SKIN. HARMFUL IF INHALED. AFFECTS TEETH. WATER REACTIVE. CANCER HAZARD. STRONG INORGANIC ACID MISTS CONTAINING SULFURIC ACID CAN CAUSE CANCER. Risk of cancer depends on duration and level of exposure.
Label Precautions:
Do not get in eyes, on skin, or on clothing.
Do not breathe mist.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Do not contact with water.
Label First Aid:
In all cases call a physician immediately. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before re-use. Excess acid on skin can be neutralized with a 2% bicarbonate of soda solution. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 8.
Disclaimer:
****

Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS

INFORMATION.
****

Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

ATTACHMENT 22.4

Please reduce your browser font size for better viewing and printing.

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 202-483-7616

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

MALLINCKRODT J.T.Baker

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

SODIUM HYDROXIDE

MSDS Number: S4034 --- *Effective Date: 08/20/98*

1. Product Identification

Synonyms: Caustic soda; lye; sodium hydroxide solid; sodium hydrate
CAS No.: 1310-73-2
Molecular Weight: 40.00
Chemical Formula: NaOH
Product Codes:
J.T. Baker: 3718, 3721, 3722, 3723, 3728, 3734, 3736, 5045, 5565
Mallinckrodt: 7001, 7680, 7708, 7712, 7772, 7798

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Sodium Hydroxide	1310-73-2	99 - 100%	Yes

3. Hazards Identification

Emergency Overview

POISON! DANGER! CORROSIVE. MAY BE FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES BURNS TO ANY AREA OF CONTACT. REACTS WITH WATER, ACIDS AND OTHER MATERIALS.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Poison)
Flammability Rating: 0 - None
Reactivity Rating: 2 - Moderate
Contact Rating: 4 - Extreme (Corrosive)
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES
Storage Color Code: White Stripe (Store Separately)

Potential Health Effects

Inhalation:
Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Severe pneumonitis may occur.
Ingestion:
Corrosive! Swallowing may cause severe burns of mouth, throat, and stomach. Severe scarring of tissue and death may result. Symptoms may include bleeding, vomiting, diarrhea, fall in blood pressure. Damage may appears days after exposure.
Skin Contact:
Corrosive! Contact with skin can cause irritation or severe burns and scarring with greater exposures.
Eye Contact:
Corrosive! Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Chronic Exposure:
Prolonged contact with dilute solutions or dust has a destructive effect upon tissue.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance.

---

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:
DO NOT INDUCE VOMITING! Give large quantities of water or milk if available. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:
Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Call a physician, immediately. Wash clothing before reuse.
Eye Contact:

Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

Note to Physician:
Perform endoscopy in all cases of suspected sodium hydroxide ingestion. In cases of severe esophageal corrosion, the use of therapeutic doses of steroids should be considered. General supportive measures with continual monitoring of gas exchange, acid-base balance, electrolytes, and fluid intake are also required.

---

5. Fire Fighting Measures

Fire:
Not considered to be a fire hazard. Hot or molten material can react violently with water. Can react with certain metals, such as aluminum, to generate flammable hydrogen gas.
Explosion:
Not considered to be an explosion hazard.
Fire Extinguishing Media:
Use any means suitable for extinguishing surrounding fire. Adding water to caustic solution generates large amounts of heat.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Keep unnecessary and unprotected people away from area of spill. Wear appropriate personal protective equipment as specified in Section 8. Spills: Pick up and place in a suitable container for reclamation or disposal, using a method that does not generate dust. Do not flush caustic residues to the sewer. Residues from spills can be diluted with water, neutralized with dilute acid such as acetic, hydrochloric or sulfuric. Absorb neutralized caustic residue on clay, vermiculite or other inert substance and package in a suitable container for disposal. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

---

7. Handling and Storage

Keep in a tightly closed container. Protect from physical damage. Store in a cool, dry, ventilated area away from sources of heat, moisture and incompatibilities. Always add the caustic to water while stirring; never the reverse. Containers of this material may be hazardous when empty since they retain product residues (dust, solids); observe all warnings and precautions listed for the product. Do not store with aluminum or magnesium. Do not mix with acids or organic materials.

ATTACHMENT 22.4

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
- OSHA Permissible Exposure Limit (PEL):
2 mg/m3 Ceiling
- ACGIH Threshold Limit Value (TLV):
2 mg/m3 Ceiling

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, a half-face dust/mist respirator may be worn for up to ten times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. A full-face piece dust/mist respirator may be worn up to 50 times the exposure limit, or the maximum use concentration specified by the appropriate regulatory agency, or respirator supplier, whichever is lowest. For emergencies or instances where the exposure levels are not known, use a full-facepiece positive-pressure, air-supplied respirator.
WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.

Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

Appearance:
White, deliquescent pellets or flakes.

Odor:
Odorless.

Solubility:
111 g/100 g of water.

Specific Gravity:
2.13

pH:
13 - 14 (0.5% soln.)

% Volatiles by volume @ 21C (70F):
0

Boiling Point:
1390C (2534F)
Melting Point:
318C (604F)
Vapor Density (Air=1):
> 1.0
Vapor Pressure (mm Hg):
Negligible.
Evaporation Rate (BuAc=1):
No information found.

---

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Very hygroscopic. Can slowly pick up moisture from air and react with carbon dioxide from air to form sodium carbonate.
Hazardous Decomposition Products:
Sodium oxide. Decomposition by reaction with certain metals releases flammable and explosive hydrogen gas.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Sodium hydroxide in contact with acids and organic halogen compounds, especially trichloroethylene, may causes violent reactions. Contact with nitromethane and other similar nitro compounds causes formation of shock-sensitive salts. Contact with metals such as aluminum, magnesium, tin, and zinc cause formation of flammable hydrogen gas. Sodium hydroxide, even in fairly dilute solution, reacts readily with various sugars to produce carbon monoxide. Precautions should be taken including monitoring the tank atmosphere for carbon monoxide to ensure safety of personnel before vessel entry.
Conditions to Avoid:
Moisture, dusting and incompatibles.

---

11. Toxicological Information

Irritation data: skin, rabbit: 500 mg/24H severe; eye rabbit: 50 ug/24H severe; investigated as a mutagen.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Sodium Hydroxide (1310-73-2)	No	No	None

---

12. Ecological Information

Environmental Fate:
No information found.
Environmental Toxicity:
No information found.

---

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

---

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: SODIUM HYDROXIDE, SOLID
Hazard Class: 8
UN/NA: UN1823
Packing Group: II
Information reported for product/size: 300LB

International (Water, I.M.O.)

Proper Shipping Name: SODIUM HYDROXIDE, SOLID
Hazard Class: 8
UN/NA: UN1823
Packing Group: II
Information reported for product/size: 300LB

---

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Sodium Hydroxide (1310-73-2)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

		--Canada--		
Ingredient	Korea	DSL	NDSL	Phil.
Sodium Hydroxide (1310-73-2)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\----------------

	-SARA 302-	------SARA 313------

ATTACHMENT 22.4

Ingredient	RQ	TPQ	List	Chemical Catg.
Sodium Hydroxide (1310-73-2)	No	No	Yes	No

\Federal, State & International Regulations - Part 2\

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Sodium Hydroxide (1310-73-2)	1000	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: No Fire: No Pressure: No
Reactivity: Yes (Pure / Solid)

Australian Hazchem Code: 2R
Poison Schedule: S6
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **3** Flammability: **0** Reactivity: **1**
Label Hazard Warning:
POISON! DANGER! CORROSIVE. MAY BE FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES BURNS TO ANY AREA OF CONTACT. REACTS WITH WATER, ACIDS AND OTHER MATERIALS.
Label Precautions:
Do not get in eyes, on skin, or on clothing.
Do not breathe dust.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Label First Aid:
If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen. In all cases get medical attention immediately.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 10.
Disclaimer:
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Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.

****

Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

ATTACHMENT 22.5

Please reduce your browser font size for better viewing and printing.

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

MALLINCKRODT J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 202-483-7616

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

SODIUM SULFATE ANHYDROUS

MSDS Number: S5018 --- *Effective Date: 03/01/00*

1. Product Identification

Synonyms: Disodium sulfate; sodium sulfate anhydrous; sulfuric acid, disodium salt; sodium sulfate
CAS No.: 7757-82-6
Molecular Weight: 142.04
Chemical Formula: Na2 SO4
Product Codes:
J.T. Baker: 3375, 3887, 3891, 3898, 3902
Mallinckrodt: 6775, 7803, 8016, 8020, 8022, 8024, 8025, 8026, 8028, 9675

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Sodium Sulfate	7757-82-6	90 - 100%	Yes

3. Hazards Identification

Emergency Overview

As part of good industrial and personal hygiene and safety procedure, avoid all unnecessary exposure to the chemical substance and ensure prompt removal from skin, eyes and clothing.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 0 - None
Flammability Rating: 0 - None
Reactivity Rating: 0 - None
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT
Storage Color Code: Orange (General Storage)

Potential Health Effects

Inhalation:
Not expected to be a health hazard.
Ingestion:
Mildly toxic by ingestion. Slowly absorbed from the alimentary tract. Because of osmotic activity, it will draw water into the lumen of the bowel and may cause purging, fluid loss, blood in stools, fall of blood pressure, and high sodium levels in the blood.
Skin Contact:
No adverse effects expected.
Eye Contact:
No adverse effects expected but dust may cause mechanical irritation.
Chronic Exposure:
No information found.
Aggravation of Pre-existing Conditions:
No information found.

4. First Aid Measures

Inhalation:
Not expected to require first aid measures.
Ingestion:
Give several glasses of water to drink to dilute. If large amounts were swallowed, get medical advice.
Skin Contact:
Wash exposed area with soap and water. Get medical advice if irritation develops.
Eye Contact:
Wash thoroughly with running water. Get medical advice if irritation develops.

5. Fire Fighting Measures

Fire:
Not considered to be a fire hazard.

Explosion:
Not considered to be an explosion hazard, but violent explosions occur when potassium sulfate and sodium sulfate are melted with aluminum.
Fire Extinguishing Media:
Use any means suitable for extinguishing surrounding fire.
Special Information:
Use protective clothing and breathing equipment appropriate for the surrounding fire.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Spills: Sweep up and containerize for reclamation or disposal. Vacuuming or wet sweeping may be used to avoid dust dispersal.

---

7. Handling and Storage

Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect against physical damage. Isolate from incompatible substances. Containers of this material may be hazardous when empty since they retain product residues (dust, solids); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
None established.
Ventilation System:
In general, dilution ventilation is a satisfactory health hazard control for this substance. However, if conditions of use create discomfort to the worker, a local exhaust system should be considered.
Personal Respirators (NIOSH Approved):
For conditions of use where exposure to the dust or mist is apparent, a half-face dust/mist respirator may be worn. For emergencies or instances where the exposure levels are not known, use a full-face positive-pressure, air-supplied respirator. WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.
Skin Protection:
Wear protective gloves and clean body-covering clothing.
Eye Protection:
Use chemical safety goggles. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

Appearance:
Fine white crystalline granules.
Odor:
Odorless.
Solubility:
Soluble in approx. 3.6 parts water, 1 in 2 parts maximum @ 33C (91F).
Density:
2.68
pH:
No information found.
% Volatiles by volume @ 21C (70F):
0
Boiling Point:
No information found.
Melting Point:
844C (1551F)
Vapor Density (Air=1):
No information found.
Vapor Pressure (mm Hg):
No information found.
Evaporation Rate (BuAc=1):
No information found.

---

10. Stability and Reactivity

Stability:
Stable in tightly closed containers under normal conditions of storage.
Hazardous Decomposition Products:
Oxides of sulfur and sodium may form when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
In combination with sodium sulfate, aluminum and magnesium will explode @ 800C (1472F); strong mineral acids and bases.
Conditions to Avoid:
Air, moisture, and incompatibles.

---

11. Toxicological Information

No LD50/LC50 information found relating to normal routes of occupational exposure.
Investigated as a tumorigen, mutagen, reproductive effector.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category

Sodium Sulfate (7757-82-6)	No	No	None

12. Ecological Information

Environmental Fate:
When released into the soil, this material is expected to leach into groundwater. This material is not expected to significantly bioaccumulate.
Environmental Toxicity:
This material is not expected to be toxic to aquatic life. The LC50/96-hour values for fish are over 100 mg/l. The EC50/48-hour values for daphnia are over 100 mg/l.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be managed in an appropriate and approved waste disposal facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Not regulated.

15. Regulatory Information

\Chemical Inventory Status - Part 1\

Ingredient	TSCA	EC	Japan	Australia
Sodium Sulfate (7757-82-6)	Yes	Yes	Yes	Yes

\Chemical Inventory Status - Part 2\

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Sodium Sulfate (7757-82-6)	Yes	Yes	No	Yes

\Federal, State & International Regulations - Part 1\

Ingredient	-SARA 302- RQ	TPQ	List	-----SARA 313----- Chemical Catg.
Sodium Sulfate (7757-82-6)	No	No	No	No

\Federal, State & International Regulations - Part 2\

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Sodium Sulfate (7757-82-6)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: No Chronic: No Fire: No Pressure: No
Reactivity: No (Pure / Solid)

Australian Hazchem Code: No information found.
Poison Schedule: No information found.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **1** Flammability: **0** Reactivity: **0**
Label Hazard Warning:
As part of good industrial and personal hygiene and safety procedure, avoid all unnecessary exposure to the chemical substance and ensure prompt removal from skin, eyes and clothing.
Label Precautions:
None.
Label First Aid:
Not applicable.
Product Use:
Laboratory Reagent.
Revision Information:
No changes.
Disclaimer:
****

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Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

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MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

MALLINCKRODT J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

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Chemtrec: 202-483-7616

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

METHYL ALCOHOL

MSDS Number: M2015 --- *Effective Date: 12/14/00*

1. Product Identification

Synonyms: Wood alcohol; methanol; carbinol
CAS No.: 67-56-1
Molecular Weight: 32.04
Chemical Formula: CH3OH
Product Codes:
J.T. Baker: 5217, 5370, 5794, 5807, 5811, 5842, 5869, 9049, 9063, 9065, 9066, 9067, 9069, 9070, 9071, 9073, 9075, 9076, 9077, 9091, 9093, 9096, 9097, 9098, 9263, 9893
Mallinckrodt: 3004, 3006, 3016, 3017, 3018, 3024, 3041, 3701, 4295, 5160, 8814, H080, H488, H603, H985, V079, V571

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Methyl Alcohol	67-56-1	100%	Yes

3. Hazards Identification

Emergency Overview

POISON! DANGER! VAPOR HARMFUL. MAY BE FATAL OR CAUSE BLINDNESS IF SWALLOWED. HARMFUL IF INHALED OR ABSORBED THROUGH SKIN. CANNOT BE MADE NONPOISONOUS. FLAMMABLE

ATTACHMENT 22.6

LIQUID AND VAPOR. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM AND LIVER.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Poison)
Flammability Rating: 4 - Extreme (Flammable)
Reactivity Rating: 1 - Slight
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

Inhalation:
A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Once absorbed into the body, it is very slowly eliminated. Symptoms of overexposure may include headache, drowsiness, nausea, vomiting, blurred vision, blindness, coma, and death. A person may get better but then worse again up to 30 hours later.

Ingestion:
Toxic. Symptoms parallel inhalation. Can intoxicate and cause blindness. Usual fatal dose: 100-125 milliliters.

Skin Contact:
Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure.

Eye Contact:
Irritant. Continued exposure may cause eye lesions.

Chronic Exposure:
Marked impairment of vision has been reported. Repeated or prolonged exposure may cause skin irritation.

Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired liver or kidney function may be more susceptible to the effects of the substance.

---

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

Ingestion:
Induce vomiting immediately as directed by medical personnel. Never give anything by

mouth to an unconscious person. Get medical attention immediately.

Skin Contact:

Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.

Eye Contact:

Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

---

5. Fire Fighting Measures

Fire:

Flash point: 12C (54F) CC
Autoignition temperature: 464C (867F)
Flammable limits in air % by volume:
lel: 6.0; uel: 36
Flammable Liquid and Vapor!

Explosion:

Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Moderate explosion hazard and dangerous fire hazard when exposed to heat, sparks or flames. Sensitive to static discharge.

Fire Extinguishing Media:

Use alcohol foam, dry chemical or carbon dioxide. (Water may be ineffective.)

Special Information:

In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Use water spray to blanket fire, cool fire exposed containers, and to flush non-ignited spills or vapors away from fire. Vapors can flow along surfaces to distant ignition source and flash back.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB(R) solvent adsorbent is recommended for spills of this product.

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7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product. Do Not attempt to clean empty containers since residue is difficult to remove. Do not pressurize, cut, weld, braze, solder, drill, grind or expose such containers to heat, sparks, flame, static electricity or other sources of ignition: they may explode and cause injury or death.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
For Methyl Alcohol:
- OSHA Permissible Exposure Limit (PEL):
200 ppm (TWA)
- ACGIH Threshold Limit Value (TLV):
200 ppm (TWA), 250 ppm (STEL) skin

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details. Use explosion-proof equipment.

Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. This substance has poor warning properties.

Skin Protection:
Rubber or neoprene gloves and additional protection including impervious boots, apron, or coveralls, as needed in areas of unusual exposure.

Eye Protection:
Use chemical safety goggles. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

Appearance:
Clear, colorless liquid.

Odor:
Characteristic odor.

Solubility:
Miscible in water.
Specific Gravity:
0.8
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100
Boiling Point:
64.5C (147F)
Melting Point:
-98C (-144F)
Vapor Density (Air=1):
1.1
Vapor Pressure (mm Hg):
97 @ 20C (68F)
Evaporation Rate (BuAc=1):
5.9

---

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products:
May form carbon dioxide, carbon monoxide, and formaldehyde when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Strong oxidizing agents such as nitrates, perchlorates or sulfuric acid. Will attack some forms of plastics, rubber, and coatings. May react with metallic aluminum and generate hydrogen gas.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

---

11. Toxicological Information

Methyl Alcohol (Methanol) Oral rat LD50: 5628 mg/kg; inhalation rat LC50: 64000 ppm/4H; skin rabbit LD50: 15800 mg/kg; Irritation data-standard Draize test: skin, rabbit: 20mg/24 hr. Moderate; eye, rabbit: 100 mg/24 hr. Moderate. Investigated as a mutagen, reproductive effector.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category

Methyl Alcohol (67-56-1)	No	No	None

12. Ecological Information

Environmental Fate:
When released into the soil, this material is expected to readily biodegrade. When released into the soil, this material is expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into the water, this material is expected to have a half-life between 1 and 10 days. When released into water, this material is expected to readily biodegrade. When released into the air, this material is expected to exist in the aerosol phase with a short half-life. When released into the air, this material is expected to be readily degraded by reaction with photochemically produced hydroxyl radicals. When released into air, this material is expected to have a half-life between 10 and 30 days. When released into the air, this material is expected to be readily removed from the atmosphere by wet deposition.
Environmental Toxicity:
This material is expected to be slightly toxic to aquatic life.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: METHANOL
Hazard Class: 3
UN/NA: UN1230
Packing Group: II
Information reported for product/size: 350LB

International (Water, I.M.O.)

Proper Shipping Name: METHANOL
Hazard Class: 3.2, 6.1
UN/NA: UN1230
Packing Group: II
Information reported for product/size: 350LB

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Methyl Alcohol (67-56-1)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Methyl Alcohol (67-56-1)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\--------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Methyl Alcohol (67-56-1)	No	No	Yes	No

--------\Federal, State & International Regulations - Part 2\--------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Methyl Alcohol (67-56-1)	5000	U154	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: Yes Pressure: No
Reactivity: No (Pure / Liquid)

Australian Hazchem Code: 2PE
Poison Schedule: S6
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
POISON! DANGER! VAPOR HARMFUL. MAY BE FATAL OR CAUSE BLINDNESS IF SWALLOWED. HARMFUL IF INHALED OR ABSORBED THROUGH SKIN. CANNOT BE MADE NONPOISONOUS. FLAMMABLE LIQUID AND VAPOR. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM AND LIVER.
Label Precautions:
Avoid breathing vapor.
Avoid contact with eyes, skin and clothing.
Wash thoroughly after handling.

Keep container closed.
Use only with adequate ventilation.
Keep away from heat, sparks and flame.

Label First Aid:

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. If swallowed, induce vomiting immediately as directed by medical personnel. Never give anything by mouth to an unconscious person. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. In all cases get medical attention immediately.

Product Use:

Laboratory Reagent.

Revision Information:

MSDS Section(s) changed since last revision of document include: 5.

Disclaimer:

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****

Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

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MALLINCKRODT J.T.Baker

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CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 202-483-7616

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

ACETONE

MSDS Number: A0446 --- *Effective Date: 04/09/98*

1. Product Identification

Synonyms: Dimethylketone; 2-propanone; dimethylketal
CAS No.: 67-64-1
Molecular Weight: 58.08
Chemical Formula: (CH3)2CO
Product Codes:
J.T. Baker: 5356, 5580, 5805, 9001, 9002, 9003, 9004, 9005, 9006, 9007, 9008, 9009, 9010, 9015, 9036, 9125, 9254, 9271, A134
Mallinckrodt: 0018, 2432, 2435, 2437, 2438, 2440, 2443, 2445, 2850, H451, H580, H981

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Acetone	67-64-1	99 - 100%	Yes

3. Hazards Identification

Emergency Overview

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL IF SWALLOWED OR INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS

CENTRAL NERVOUS SYSTEM.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 1 - Slight
Flammability Rating: 4 - Extreme (Flammable)
Reactivity Rating: 2 - Moderate
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

Inhalation:
Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache. Higher concentrations can produce central nervous system depression, narcosis, and unconsciousness.
Ingestion:
Swallowing small amounts is not likely to produce harmful effects. Ingestion of larger amounts may produce abdominal pain, nausea and vomiting. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms are expected to parallel inhalation.
Skin Contact:
Irritating due to defatting action on skin. Causes redness, pain, drying and cracking of the skin.
Eye Contact:
Vapors are irritating to the eyes. Splashes may cause severe irritation, with stinging, tearing, redness and pain.
Chronic Exposure:
Prolonged or repeated skin contact may produce severe irritation or dermatitis.
Aggravation of Pre-existing Conditions:
Use of alcoholic beverages enhances toxic effects. Exposure may increase the toxic potential of chlorinated hydrocarbons, such as chloroform, trichloroethane.

---

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.
Ingestion:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately.

Skin Contact:
Immediately flush skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Get medical attention. Wash clothing before reuse. Thoroughly clean shoes before reuse.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting upper and lower eyelids occasionally. Get medical attention.

---

5. Fire Fighting Measures

Fire:
Flash point: -20C (-4F) CC
Autoignition temperature: 465C (869F)
Flammable limits in air % by volume:
lel: 2.5; uel: 12.8
Extremely Flammable Liquid and Vapor! Vapor may cause flash fire.
Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Vapors can flow along surfaces to distant ignition source and flash back. Contact with strong oxidizers may cause fire. Sealed containers may rupture when heated. This material may produce a floating fire hazard. Sensitive to static discharge.
Fire Extinguishing Media:
Dry chemical, alcohol foam or carbon dioxide. Water may be ineffective. Water spray may be used to keep fire exposed containers cool, dilute spills to nonflammable mixtures, protect personnel attempting to stop leak and disperse vapors.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

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6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB(R) solvent adsorbent is recommended for spills of this product.

---

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Separate from incompatibles. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment, including explosion proof ventilation. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

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8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
Acetone:
-OSHA Permissible Exposure Limit (PEL):
1000 ppm (TWA)

-ACGIH Threshold Limit Value (TLV):
500 ppm (TWA), 750 ppm (STEL) A4 - not classifiable as a human carcinogen
Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, a half-face organic vapor respirator may be worn for up to ten times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. A full-face piece organic vapor respirator may be worn up to 50 times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. For emergencies or instances where the exposure levels are not known, use a full-face piece positive-pressure, air-supplied respirator.
WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.
Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.
Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

---

9. Physical and Chemical Properties

Appearance:
Clear, colorless, volatile liquid.
Odor:
Fragrant, mint-like
Solubility:
Miscible in all proportions in water.
Specific Gravity:
0.79 @ 20C/4C
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100
Boiling Point:
56.5C (133F) @ 760 mm Hg
Melting Point:
-95C (-139F)
Vapor Density (Air=1):
2.0
Vapor Pressure (mm Hg):
400 @ 39.5C (104F)
Evaporation Rate (BuAc=1):
ca. 7.7

---

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products:
Carbon dioxide and carbon monoxide may form when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Concentrated nitric and sulfuric acid mixtures, oxidizing materials, chloroform, alkalis, chlorine compounds, acids, potassium t-butoxide.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

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11. Toxicological Information

Oral rat LD50: 5800 mg/kg; Inhalation rat LC50: 50,100mg/m3; Irritation eye rabbit, Standard Draize, 20 mg severe; investigated as a tumorigen, mutagen, reproductive effector.

--------\Cancer Lists\----------

---NTP Carcinogen---

Ingredient	Known	Anticipated	IARC Category
Acetone (67-64-1)	No	No	None

12. Ecological Information

Environmental Fate:
When released into the soil, this material is expected to readily biodegrade. When released into the soil, this material is expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material is expected to readily biodegrade. When released to water, this material is expected to quickly evaporate. This material has a log octanol-water partition coefficient of less than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material may be moderately degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material may be moderately degraded by photolysis. When released into the air, this material is expected to be readily removed from the atmosphere by wet deposition.
Environmental Toxicity:
This material is not expected to be toxic to aquatic life. The LC50/96-hour values for fish are over 100 mg/l.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: ACETONE
Hazard Class: 3
UN/NA: UN1090
Packing Group: II
Information reported for product/size: 350LB

International (Water, I.M.O.)

Proper Shipping Name: ACETONE
Hazard Class: 3.1
UN/NA: UN1090

Packing Group: II
Information reported for product/size: 350LB

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\--------

Ingredient	TSCA	EC	Japan	Australia
Acetone (67-64-1)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\--------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Acetone (67-64-1)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\--------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Acetone (67-64-1)	No	No	Yes	No

--------\Federal, State & International Regulations - Part 2\--------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Acetone (67-64-1)	5000	U002	No

Chemical Weapons Convention: No TSCA 12(b): Yes CDTA: Yes
SARA 311/312: Acute: Yes Chronic: No Fire: Yes Pressure: No
Reactivity: No (Pure / Liquid)

Australian Hazchem Code: 2[Y]E
Poison Schedule: No information found.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL IF SWALLOWED OR INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS CENTRAL NERVOUS SYSTEM.
Label Precautions:
Keep away from heat, sparks and flame.

Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor.
Avoid contact with eyes, skin and clothing.

Label First Aid:

Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. In all cases, get medical attention.

Product Use:

Laboratory Reagent.

Revision Information:

MSDS Section(s) changed since last revision of document include: 8.

Disclaimer:

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Prepared by: Strategic Services Division
Phone Number: (314) 539-1600 (U.S.A.)

MDEQ-ENVIRONMENTAL LAB
SEMI-VOLATILE ORGANIC STANDARDS

SVOC Tracking Number	Date Prepared	EL or SVOC Tracking Number	Analyte or Mix	Preparers Initials	Solvent	Solvent Tracking Number	Initial Concentration*	Initial Volume/Wt Standard*	Final Volume*	Final Concentration*	Expiration Date
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											
SE-											

ATTACHMENT 22.8

FILE:SV-STD
12/30/98

*INCLUDE UNITS

MDEQ-ENVIRONMENTAL LAB
STOCK TRACKING LOG

Environmental Lab Tracking Number	QTY (each)	Filley Tracking Number	Date Received (at lab)	Receiver Initials	Chemical,Analyte, Reagent or Solvent	Vendor and Catalog #	PO #	Manufacturer and Lot Number	Expiration Date
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
EL-									
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EL-									
EL-									
EL-									
EL-									
EL-									

ATTACHMENT 22.9

FILE:ST-STD
12/30/98

ATTACHMENT 22.10

Scan:_____
Method: 3510
Date:_____
Analyst:_____
Batch #_____

MDEQ-Environmental Lab
Water Extraction Logbook- Organics

Surrogate #_____
Spike #_____

	MeCl#		NaOH#		H2SO4#		Na2SO4#		Hexane#
#	Sample #	Collection Date	Adjusted pH	Initial Volume (mL)	Surrogate Volume (mL)	Spike Volume (mL)	Final Volume (mL)	Scan	Comments
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									

COMMENTS

ATTACHMENT 22.11

CERTIFIED WEIGHT REPORT

Part Number: 93009
Lot Number: 102903
Description: EPA Method 625 LCS Spike
54 components
Expiration Date: 102908

Nominal Concentration (µg/mL): Varied

Lot #	Solvent(s):
CI561	Methanol

Formulated By: Vincent K. Criscio, Jr. DATE 102903

Reviewed By: Pedro L. Rentas DATE 102903

5E-05 Balance Uncertainty

Volume(s) shown below were combined and diluted to : 400.0 0.050 Flask Uncertainty

Compound	Part Number	Lot Number	Dil. Factor	Initial Vol. (mL)	Uncertainty Pipette	Initial Conc.(ug/mL)	Final Conc.(ug/mL)	Expanded Uncertainty (+/-) (µg/mL)	MSDS Information (Solvent Safety Info. On Attached pg.) CAS#	OSHA PEL (TWA)	LD50
1. Acenaphthene	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00450	00083-32-9	N/A	ipr-rat 600mg/kg
2. Acenaphthylene	92987	102903	0.50	200.0	0.013	100.0	50.0	0.00450	00208-96-8	N/A	N/A
3. Anthracene	92987	102903	0.50	200.0	0.013	100.0	50.0	0.00450	00120-12-7	0.2mg/m3 (8H)	ipr-mus 430mg/kg
4. Benzo(a)anthracene	92987	102903	0.50	200.0	0.013	100.0	50.0	0.00450	00056-55-3	N/A	N/A
5. Benzo(b)fluoranthene	92987	102903	0.50	200.0	0.013	100.0	50.0	0.00450	00205-99-2	N/A	N/A
6. Benzo(k)fluoranthene	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00450	00207-08-9	N/A	orl-rat 2820mg/kg
7. Benzo(g,h,i)perylene	92987	102903	0.50	200.0	0.013	100.0	50.0	0.00450	00191-24-2	N/A	N/A
8. Benzo(a)pyrene	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00453	00050-32-8	0.2mg/m3 (8H)	scu-rat 50mg/kg
9. 4-Bromophenyl phenyl ether	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00450	00101-55-3	N/A	N/A
10. Benzyl butyl phthalate	92987	102903	0.50	200.0	0.013	100.0	50.0	0.00450	00085-68-7	N/A	orl-rat 2330mg/kg
11. bis(2-Chloroethoxy) methane	92987	102903	0.50	200.0	0.013	100.2	50.1	0.00450	00111-91-1	N/A	N/A
12. bis(2-Chloroethyl) ether	92987	102903	0.50	200.0	0.013	100.2	50.1	0.00450	00111-44-4	15 ppm (90mg/m3/8H)(ski	orl-rat 75mg/kg
13. bis(2-Chloroisopropyl) ether	92987	102903	0.50	200.0	0.013	100.2	50.1	0.01043	00108-60-1	N/A	orl-rat 240mg/kg
14. 2-Chloronaphthalene	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00450	00091-58-7	N/A	orl-rat 2078mg/kg
15. 4-Chlorophenyl phenyl ether	92987	102903	0.50	200.0	0.013	100.5	50.2	0.00450	07005-72-3	N/A	N/A
16. Chrysene	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00450	00218-01-9	0.2mg/m3	N/A
17. Di-n-butyl phthalate	92987	102903	0.50	200.0	0.013	100.1	50.1	0.00450	00084-74-2	5mg/m3/8H	orl-rat 8000mg/kg
18. Di-n-octyl phthalate	92987	102903	0.50	200.0	0.013	100.2	50.1	0.00450	00117-84-0	N/A	ipr-mus 65g/kg
19. Dibenzo(a,h)anthracene	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00450	00053-70-3	0.2mg/m3	N/A
20. 1,2-Dichlorobenzene	92987	102903	0.50	200.0	0.013	100.2	50.1	0.00450	00095-50-1	50 ppm (300mg/m3) (CL)	orl-rat 500mg/kg
21. 1,3-Dichlorobenzene	92987	102903	0.50	200.0	0.013	100.3	50.2	0.00453	00541-73-1	N/A	ipr-mus 1062mg/kg
22. 1,4-Dichlorobenzene	92987	102903	0.50	200.0	0.013	100.0	50.0	0.00450	00106-46-7	75 ppm (450mg/m3/8H)	orl-rat 500mg/kg
23. 2,4-Dinitrotoluene	92987	102903	0.50	200.0	0.013	100.0	50.0	0.00456	00121-14-2	1.5mg/m3/8H (skin)	orl-rat 268mg/kg
24. 2,6-Dinitrotoluene	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00450	00606-20-2	1.5mg/m3/8H (skin)	orl-rat 177mg/kg
25. Diethyl phthalate	92987	102903	0.50	200.0	0.013	100.2	50.1	0.00450	00084-66-2	5mg/m3/8H	orl-rat 8600mg/kg
26. Dimethyl phthalate	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00450	00131-11-3	5mg/m3/8H	orl-rat 6800mg/kg
27. bis(2-Ethylhexyl) phthalate	92987	102903	0.50	200.0	0.013	100.2	50.1	0.00450	00117-81-7	5mg/m3/8H	orl-rat 30600mg/kg
28. Fluoranthene	92987	102903	0.50	200.0	0.013	100.1	50.1	0.00453	00206-44-0	N/A	orl-rat 2000mg/kg
29. Fluorene	92987	102903	0.50	200.0	0.013	100.1	50.1	0.00453	00086-73-7	N/A	ipr-mus 2 g/kg
30. Hexachlorobenzene	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00450	00118-74-1	N/A	orl-rat 10g/kg
31. Hexachlorobutadiene	92987	102903	0.50	200.0	0.013	100.2	50.1	0.00453	00087-68-3	0.02 ppm (0.24mg/m3/8H)	orl-rat 90mg/kg
32. Hexachlorocyclopentadiene	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00453	00077-47-4	0.01 ppm (0.1mg/m3/8H)	orl-rat 1300mg/kg
33. Hexachloroethane	92987	102903	0.50	200.0	0.013	100.0	50.0	0.00450	00067-72-1	1 ppm (10mg/m3/8H)(skin	orl-rat 4460mg/kg
34. Indeno(1,2,3-cd)pyrene	92987	102903	0.50	200.0	0.013	100.1	50.1	0.00450	00193-39-5	N/A	N/A
35. Isophorone	92987	102903	0.50	200.0	0.013	100.2	50.1	0.00456	00078-59-1	25 ppm	orl-rat 2330mg/kg
36. Naphthalene	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00450	00091-20-3	10 ppm (50mg/m3/8H)	orl-rat 490mg/kg
37. Nitrobenzene	92987	102903	0.50	200.0	0.013	100.1	50.1	0.00450	00098-95-3	1 ppm (5mg/m3/8H)(skin)	orl-rat 780mg/kg
38. N-Nitrosodimethylamine	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00453	00062-75-9	N/A	orl-rat 58mg/kg
39. N-Nitrosodi-n-propylamine	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00450	00621-64-7	N/A	orl-rat 480mg/kg
40. N-Nitrosodiphenylamine	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00453	00086-30-6	N/A	orl-rat 2140mg/kg
41. Phenanthrene	92987	102903	0.50	200.0	0.013	100.1	50.1	0.00450	00085-01-8	0.2mg/m3/8H	orl-mus 700mg/kg
42. Pyrene	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00453	00129-00-0	0.2mg/m3/8H	orl-rat 2700mg/kg
43. 1,2,4-Trichlorobenzene	92987	102903	0.50	200.0	0.013	100.1	50.0	0.00450	00120-82-1	5 ppm (CL) (40mg/m3)	orl-rat 756mg/kg
44. 4-Chloro-3-methylphenol	92988	102903	0.50	200.0	0.013	200.4	100.2	0.00480	00059-50-7	N/A	orl-rat 1830mg/kg
45. 2-Chlorophenol	92988	102903	0.50	200.0	0.013	200.4	100.2	0.00480	00095-57-8	N/A	orl-rat 670mg/kg
46. 2,4-Dichlorophenol	92988	102903	0.50	200.0	0.013	200.3	100.1	0.00480	00120-83-2	N/A	orl-rat 580mg/kg
47. 2,4-Dimethylphenol	92988	102903	0.50	200.0	0.013	200.4	100.2	0.00484	00105-67-9	N/A	orl-rat 3200mg/kg
48. 2,4-Dinitrophenol	92988	102903	0.50	200.0	0.013	200.4	100.2	0.00482	00051-28-5	N/A	orl-rat 30mg/kg
49. 4,6-Dinitro-2-methylphenol	92988	102903	0.50	200.0	0.013	200.2	100.1	0.00482	00534-52-1	0.2mg/m3/8H (skin)	orl-rat 10mg/kg
50. 2-Nitrophenol	92988	102903	0.50	200.0	0.013	200.2	100.1	0.00482	00088-75-5	N/A	orl-rat 334mg/kg
51. 4-Nitrophenol	92988	102903	0.50	200.0	0.013	200.2	100.1	0.00482	00100-02-7	N/A	orl-rat 250mg/kg
52. Pentachlorophenol	92988	102903	0.50	200.0	0.013	200.1	100.0	0.00482	00087-86-5	0.5mg/m3/8H (skin)	orl-rat 27mg/kg
53. Phenol	92988	102903	0.50	200.0	0.013	200.4	100.2	0.00480	00108-95-2	5 ppm (19mg/m3/8H)(skin	orl-rat 317mg/kg
54. 2,4,6-Trichlorophenol	92988	102903	0.50	200.0	0.013	200.4	100.2	0.00480	00088-06-2	N/A	orl-rat 820mg/kg

EFFECTIVE DATE: 03/2007 SOP# 502 REVISION # 1

ORGANOCHLORINE PESTICIDES, POLYCHLORINATED BIPHENYLS (PCBS), AND CHLORINATED HYDROCARBONS BY GAS CHROMATOGRAPHY

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1.0 Applicable Analytical Methods

1.1 Organochlorine Pesticides by Gas Chromatography, U.S. Environmental Protection Agency, Method 8081A, SW846, Revision 1, December 1996.

1.2 Polychlorinated Biphenyls (PCBs) By Gas Chromatography, U.S. Environmental Protection Agency, Method 8082A, SW846, Revision 1, November 2000.

1.3 Determinative Chromatographic Separations, U.S. Environmental Protection Agency, Method 8000C, SW846, Revision 3, March 2003.

1.4 Quality Control, U.S. Environmental Protection Agency, Chapter 1, SW846, Revision 1, July 1992.

1.5 Organic Extraction and Sample Preparation, U.S. Environmental Protection Agency, Method 3500B, SW846, Revision 2, December 1996.

1.6 Cleanup, U.S. Environmental Protection Agency, Method 3600C, SW846, Revision 3, December 1996.

1.7 Organochlorine Pesticides and PCBs, U.S. Environmental Protection Agency, Method 608, 40CFR, Part 136, Appendix A.

1.8 Chlorinated Hydrocarbons, U.S. Environmental Protection Agency, Method 612, 40 CFR, Part 136, Appendix A.

2.0 Matrix or Matrices

2.1 Aqueous.

2.2 Soil.

2.3 Sediment.

2.4 Sludge.

2.5 Clay.

2.6 Oil.

2.7 Solid or liquid waste.

3.0 Method Detection Limits (MDLs) and Reporting Limits (RLs)

3.1 All MDLs shall be performed according to the Code of Federal Regulations 40 CFR, Part 136, Appendix B. See internal procedure SOP 103.

3.2 Every individual analyte to be quantitated by this method must have an MDL study performed for it. The MDL results for Aroclors 1016 and 1260 are used to determine MDLs for the rest of the PCBs. Results obtained for analytes that have not had an MDL calculated by this method must be considered estimated. MDLs are analyzed annually or whenever major instrument changes occur. MDLs are carried through the same extraction and cleanup procedures as samples are. (Refer to SOP 405, SOP 400, SOP 401, and SOP 403 for details.) Some compounds may need to be prepared at multiple levels. A Method Preparation Blank (MPB) is run with each batch.

3.2.1 Water MDLs – MDLs will be done for every pesticide analyte, for a combination of Aroclors 1016 and 1260 to cover all of the PCBs, and for toxaphene. Each of these sets may have to be done at multiple concentration levels to get the MDLs within the proper range.

3.2.2 Soil MDLs – MDLs will be done for every pesticide analyte, for a combination of Aroclors 1016 and 1260 to cover all of the PCBs, and for toxaphene. Each of these

sets may have to be done at multiple concentration levels to get the MDLs within the proper range.

3.3 A RL will be used as the minimum quantification limit for all analytes reported unless otherwise specified. The RL will always be routinely achievable and greater than the MDL. (Refer to internal procedure SOP 103 for further details.)

4.0 Scope and Application

4.1 This method provides for the GC/electron capture detector (ECD) determination of organochlorine pesticides and PCBs in aqueous, oil, and solid matrices. This method is restricted to use by (or under the supervision of) analysts who are experienced in the use of GC and who are skilled in the interpretation of chromatograms and their uses as a quantitative tool. The RLs for solid samples are reported on a dry-weight basis (Attachment 22.6).

4.2 The following compounds are analyzed by this method:

Analyte	Chem. Abstract Services Registry No.
Aldrin	309-00-2
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4
a-BHC	319-84-6
b-BHC	319-85-7
g-BHC (Lindane)	58-89-9
d-BHC	319-86-8
2,2',4,4',5,5'-Hexabromobiphenyl(BP-6)(PBB)	59080-40-9
a-Chlordane	5103-71-9
g-Chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan Sulfate	1031-07-8
Endrin	72-20-8
Endrin Aldehyde	7421-93-4
Endrin Ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3

Hexabromobenzene	87-82-1
*Hexachlorobenzene	118-74-1
*Hexachlorobutadiene	87-68-3
*Hexachlorocyclopentadiene	77-47-4
Mirex	2385-85-5
Methoxychlor	72-43-5
*Pentachlorobenzene	608-93-5
* Pentachloronitrobenzene	82-68-8
Toxaphene	8001-35-2
*1,2,3,4-Tetrachlorobenzene	634-66-2
*1,2,4,5-Tetrachlorobenzene	95-94-3

*Scan 3 water analysis only for NPDES permits

5.0 Method Summary

5.1 Water samples are extracted as in SOP 405. Water samples are cleaned up as in SOP 400 and SOP 403.

5.2 Solid samples are extracted as in SOP 401. Solid samples are cleaned up as in SOP 400.

5.3 Oil samples are prepared as in SOP 404. Oil samples are cleaned up as in SOP 400 and SOP 402.

5.4 The sample extracts are then injected onto a GC. The GC is temperature programmed to separate the analytes, which are then detected with an ECD.

5.5 The pesticide compounds are analyzed by GC/ECD with dual capillary columns. When unknown samples are analyzed by this method, a second capillary column of different stationary phase polarity confirms the identification of any single peak compound. Interpretations are performed by analyzing samples under the same conditions used for standards and by comparing retention times (RTs) of the unknown peaks to standards on each column. Quantitation is accomplished by comparing the response of the unknown to the initial calibration curve. Confirmation of the analysis may be done by GC/mass spectrometry (MS) provided the samples are sufficiently concentrated.

5.6 Multi-component compounds (PCBs and toxaphene) are analyzed by GC on a single capillary column and GC/ECD. Interpretations are performed by analyzing samples under the same conditions used for standards and comparing chromatographic patterns and RTs of the unknown peaks to standards on each column. Quantitation is accomplished by comparing the response of the unknown to the initial calibration curve. Confirmation of the analysis may be done by GC/MS provided the samples are sufficiently concentrated.

6.0 Definitions

6.1 MDL - The minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.

6.2 Surrogate Analyte (SA) - A pure analyte that is extremely unlikely to be found in any sample. The SA is added to a sample aliquot in known amounts before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method performance with each sample.

6.3 MPB - An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and surrogates that are used with samples. The MPB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

6.4 Laboratory Control Sample (LCS) - An aliquot of reagent water or other blank matrix to which known quantities of the target analytes are added in the laboratory. The LCS is analyzed and prepared exactly as a sample. Its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

6.5 Matrix Spike (MS) - An aliquot of a sample to which known quantities of the target analytes are added in the laboratory. The MS is analyzed and prepared exactly as a sample. Its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

6.6 Matrix Spike Duplicate (MSD) – An aliquot of a sample to which known quantities of the target analytes are added in the laboratory. The MSD is analyzed and prepared exactly as a sample and as the MS. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MSD corrected for background concentrations. Analyses of the MS and MSD indicate precision associated with laboratory procedures.

6.7 Initial Calibration Verification (ICV) – Second source standards used to check the accuracy of the calibration standards (CALs). This standard includes all target analytes and should be from a different vendor or a different lot number than the CALs.

6.8 Continuing Calibration Standard (CCS) – A mid-range standard to check the overall instrument performance.

6.9 Stock Standard (SS) – A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

6.10 Working Standard (WS) – A solution of an analyte(s) prepared in the laboratory from SSs and diluted as needed to prepare calibration solutions and other needed analyte solutions.

6.11 System Blank (SB) – An aliquot of hexane that has not undergone any preparation and is analyzed under the same analytical conditions as a sample. The purpose of the SB is to check for instrument contamination.

7.0 Interferences

7.1 Contamination by carryover can occur whenever high-level samples and low-level samples are sequentially analyzed. The sample syringe is rinsed with solvent between sample analyses to reduce carryover. Whenever an unusually high sample is encountered, the sample following it must be evaluated for contamination and rerun if carryover is suspected.

7.2 Interference from contaminants can occur from solvents, glassware, and other sample processing equipment. Use of pesticide quality solvents or better and solvent rinsing of all glassware should help minimize contaminants.

7.3 Compounds co-extracted with the analytes of interest can cause interference. Samples should be cleaned up to avoid interference whenever possible.

7.4 Some interference may still exist after this cleanup. This interference is identified through the analysis with the alternate column and/or GC/MS confirmation. The initial results are then reevaluated with the interference taken into consideration. When data is suspect for interference, it will be qualified.

7.5 The presence of elemental sulfur causes broad peaks that can interfere with the detection of early eluting pesticides. Sulfur can be removed by optimizing the collection window from the GPC column to eliminate collection of sulfur (SOP 400).

7.6 Oily sample matrices will often cause a raised baseline, RT shifts, and/or surrogate failure. Samples will usually need a dilution performed to minimize this interference. RLs will then be elevated appropriately. A silica gel clean-up (SOP 402) can also be effective at removing interferences.

8.0 Safety

8.1 Safety glasses are required in all designated laboratory areas.

8.2 Be familiar with the Laboratory Chemical Hygiene Plan.

8.3 Be familiar with the Laboratory Safety Policy (internal procedure SOP 100).

8.4 Gas cylinders must be secured to a wall or to a countertop.

8.5 Hexane material safety data sheets (MSDSs) are attached (Attachment 22.1).

8.6 Protective gloves must be worn when handling solvents.

9.0 Equipment and Supplies

9.1 Microsyringes – Various sizes.

9.2 Volumetric flasks - Class A, with ground-glass stoppers.

9.3 Vials - 2 mL autosampler vials, with crimp caps.

9.4 Pipettes - Class A.

9.5 Pasteur pipettes – Disposable.

9.6 Pipette bulbs.

9.7 Refrigerator and freezer. (SOP 128)

9.8 GC

9.8.1 Hewlett Packard model 5890 or 6890 GC - Equipped with an autosampler and split/splitless capillary injector. The instrument may have an electronic pressure controller also. The electronic pressure controller maintains constant flow as the GC temperature increases during programming.

9.8.2 Typical operating conditions can be found in Attachment 22.7. Changes or deviations can be found in the GC maintenance logbooks.

9.9 ECD – Because of its high sensitivity, the ECD should never be used without moisture and chemical traps that are in good condition installed in the carrier and make-up gas lines. It is important to create calibrations for all compounds and evaluate detector response on a regular basis.

9.9.1 Agilent ECD - For model 5890 GC.
9.9.2 Agilent micro-ECD - For model 6890 GC.

9.10 GC interface

9.10.1 The capillary column in the GC is directly interfaced into the ECD.
9.10.2 Capillary columns
9.10.3 Narrow bore capillary column – Phenomenex Zebron ZB-5, 30 m long x 0.25 mm ID x 0.25 – 1.0 µm film thickness or equivalent.
9.10.4 Narrow bore capillary column – Phenomenex Zebron ZB-35, 30 m long x 0.25 mm ID x 0.25 -1.0 µm film thickness or equivalent.

9.11 Data system - A personal computer is interfaced to the GC system. The computer system is equipped with an assortment of disk drives and a hard drive and is installed

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on a network with CD back-up capabilities. The software, TC Workstation (Turbo Chrom or Total Chrom Workstation, depending upon version) is made by P.E. Nelson and is multitasking and window driven. The software has a function for the comparison of chromatograms (Attachment 22.8).

10.0 Reagents and Standards

10.1 All reagents, solvents, and standards must be traceable to the stock inventory tracking log.

10.2 All reagents, solvents, and standards must be labeled with: date received, date opened, expiration date, tracking number, and receiver's initials.

10.3 All prepared reagents and standards must be labeled with: date prepared, expiration date, preparer's initials, tracking number, diluent, and description.

10.4 All standard logbooks must be completely filled out.

10.5 All certificates of analysis must include the stock inventory tracking number that was assigned to the standard. The Laboratory's Quality Assurance/Quality Control (QA/QC) Officer keeps original certificates.

10.6 Hydrogen (carrier gas) – Ultra-high purity.

10.7 Nitrogen (make-up gas) - Ultra-high purity.

10.8 Hexane - Pesticide grade demonstrated to be free of analytes (Attachment 22.1).

10.9 SSs - All standards must be certified reference materials. Manufacturers' certificates of analysis are retained by the Laboratory and kept as part of the overall QA/QC requirements. All SSs must be given a unique lab tracking number and logged into the SS tracking logbook (Attachment 22.3).

10.9.1 SSs are purchased as mixes in sealed ampules and are stored in a freezer at a temperature lower than -10°C.

10.9.2 CALs

10.9.2.1 Pesticide and chlorinated hydrocarbon mixes (1000 µg/mL) in hexane - Absolute Standards, part #93238 and part #93237 or equivalent. (Refer to Attachment 22.2 for compound lists.)

10.9.2.2 PCB mix (1000 µg/mL) in hexane - Absolute Standards, part #20064 or equivalent.
Aroclor 1016
Aroclor 1260

10.9.2.3 Surrogate mix (200 µg/mL) in acetone - Absolute Standards, part #20023 or equivalent.
2,4,5,6–tetrachloro-m-xylene
decachlorobiphenyl

10.9.2.4 Individual Aroclor standards (1000 µg/mL) in hexane

Aroclor	Absolute Standards Part #
1221	90124
1232	90125
1242	90126
1248	90127
1254	90128
1262	EPC00728 (EM Science)
1268	EPC00994 (EM Science)

10.9.2.5 Toxaphene standard (1000 µg/mL) in hexane - EM Science, part #EPP00388 or equivalent.

10.9.3 ICV

10.9.3.1 Pesticide mix (1000 µg/mL) - Absolute Standards, part #93238 and part #93237 or equivalent. (Refer to Attachment 22.2 for compound lists.) Different vendor or lot number than CALs.

10.9.3.2 PCB (1000 ug/mL) - Absolute Standards, part #20064 or equivalent.
Aroclor 1016
Aroclor 1260
Different vender or lot number than CALs.

10.9.4 DDT/endrin breakdown standard – Endrin 1000 µg/L, DDT 2000 µg/L in hexane from Ultra Scientific part #ISM-450 or equivalent.

10.10 Intermediate and WSs - All prepared standards must be given a unique laboratory tracking number and must be logged into the semivolatile standards logbook (Attachment 22.4). Store intermediate and working solutions at -10°C to -20°C in amber bottles with teflon-lined caps. The intermediate standards must be checked frequently for signs of degradation or evaporation. Fresh standards should be prepared if a check standard evaluation exceeds a 15% drift. All standards are good for up to six months.

10.10.1 Intermediate and working CALs – Place approximately 50 mL of hexane into the appropriate volumetric flask. Inject the SS or intermediate listed directly into the hexane. Bring to volume with hexane. Cap. Mix. Transfer into an amber bottle. Cap each bottle securely. Label. Store in a freezer at <-10°C.

		SS or Intermediate	mL	Final Volume (mL)	Final Conc (µg/L)
10.10.1.1	PESTICIDE WSs	10.9.2.1 (each)	0.125	500	250
		250 µg/L	100	200	125
		250 µg/L	40	200	50
		250 µg/L	12	100	30
		250 µg/L	8	200	10
		250 µg/L	2	100	5
		250 µg/L	1	100	2.5

		SS or Intermediate	mL	Final Volume (mL)	Final Conc (µg/L)
10.10.1.2	PCB WSs	10.9.2.2/10.9.2.3	1/.625	500	2000/250
		2000/250	100	200	1000/125
		2000/250	50	200	500/62.5
		2000/250	20	200	200/25
		2000/250	10	200	100/12.5
		2000/250	5	200	50/6.25
		1000/125	5	200	25/3.12

		SS or Intermediate	mL	Final Volume (mL)	Final Conc (µg/L)
10.10.1.3	TOXAPHENE WSs	10.9.2.5	0.2	100	2000
		10.9.2.5	0.1	100	1000
		2000	25	100	500
		2000	10	100	200
		2000	5	100	100
		1000	5	100	50
		500	5	100	25

10.10.2 ICV WSs – ICV WSs are stored in amber bottles with teflon–lined caps at less than -10°C.

10.10.2.1 100 µg/L pesticide ICV – Place 20 µL each of pesticide and chlorinated hydrocarbon ICV SSs (Section 10.9.3.1) into a 200 mL volumetric flask containing approximately 100 mL of hexane. Bring to volume with hexane. Mix. Transfer to an amber bottle. Label. Store in a freezer.

10.10.2.2 500 µg/L PCB ICV – Place 100 µL of stock (Section 10.9.3.2) into a 200 mL volumetric flask containing approximately 100 mL of hexane. Bring to volume with hexane. Mix. Transfer to an amber bottle. Label. Store in a freezer.

10.10.3 DDT/endrin (100 µg/L /50 µg/L) breakdown WS - Place 2.5 mL (Section 10.9.4) into a 50 mL volumetric flask containing 25 mL of hexane. Bring to volume with hexane. Mix. Transfer to an amber bottle. Label. Store in a freezer.

10.10.4 Single point Aroclor WSs – Place approximately 50 mL of hexane into the appropriate volumetric flask. Inject the SS or intermediate listed into the hexane. Dilute to the mark. Cap. Mix. Transfer into an amber bottle. Cap. Label. Store in a freezer at <-10°C.

10.10.4.1 Aroclor 1221/1262 mix (2000 µg/L) – Place 200 µL of Aroclor 1221 and 200 µL of Aroclor 1262 (Section 10.9.2.4) into a 100 mL volumetric flask. Dilute. Mix.

10.10.4.1.1 Aroclor 1221/1262 WS (500 µg/L) – Place 25 mL of the mixture from Section 10.10.4.1 into a 100 mL volumetric flask. Dilute. Mix.

10.10.4.2 Aroclor 1232/1268 mix (2000 µg/L) – Place 200 µL of Aroclor 1232 and 200 µL of Aroclor 1262 (Section 10.9.2.4) into a 100 mL volumetric flask. Dilute. Mix.

10.10.4.2.1 Aroclor 1232/1268 WS (500 µg/L) – Place 25 mL of the mixture from Section 10.10.4.2 into a 100 mL volumetric flask. Dilute. Mix.

10.10.4.3 Aroclor 1242 (2000 µg/L) – Place 200 µL of Aroclor 1242 (Section 10.9.2.4) into a 100 mL volumetric flask. Dilute. Mix.

10.10.4.3.1 Aroclor 1242 WS (500 µg/L) – Place 25 mL of the mixture from Section 10.10.4.3 into a 100 mL volumetric flask. Dilute. Mix.

10.10.4.4 Aroclor 1248 (2000 µg/L) – Place 200 µL of Aroclor 1248 (Section 10.9.2.4) into a 100 mL volumetric flask. Dilute. Mix.

10.10.4.4.1 Aroclor 1248 WS (500 µg/L) – Place 25 mL of the mixture from Section 10.10.4.4 into a 100 mL volumetric flask. Dilute. Mix.

10.10.4.5 Aroclor 1254 (2000 µg/L) - Place 200 µL of Aroclor 1254 (Section 10.9.2.4) into a 100 mL volumetric flask. Dilute. Mix.

10.10.4.5.1 Aroclor 1254 WS (500 µg/L) – Place 25 mL of the mixture from Section 10.10.4.5 into a 100 mL volumetric flask. Dilute. Mix.

11.0 Sample Collection, Preservation, and Handling

11.1 Aqueous samples – Refer to SOP 405.

11.2 Solid samples – Refer to SOP 401.

11.3 Waste samples – Refer to SOP 404.

11.4 Holding times – Sample extracts may be analyzed for up to 40 days from the extraction date.

11.5 Storage – Sample extracts must be stored in a refrigerator or freezer, protected from light, in crimp cap vials or test tubes with teflon-lined caps, until analysis.

12.0 Quality Control (QC)

12.1 DDT/endrin breakdown analysis - Prior to the analyses of any samples, blanks, CALs, or QA/QC samples for pesticides, the GC system must meet the acceptance criteria for the instrument performance check solution containing DDT/endrin. GC injector

ports can be of critical concern, especially in the analysis of DDT and endrin. Injectors that are contaminated, chemically active, or too hot can cause the degradation (breakdown) of the analytes. Endrin and DDT break down to endrin aldehyde and/or endrin ketone and DDD and/or DDE respectively. If the DDT or endrin compounds in the CCS are low, excessive breakdown should be suspected. When such breakdown is observed, corrective actions are to clean and deactivate the injector port and/or break off up to 30 cm of the column and remount it. The injector temperature should be checked and lowered if indicated.

12.1.1 Inject 2 µL of a solution containing DDT at 100 µg/L and endrin at 50 µg/L (Section 10.10.3) into the GC.

12.1.2 See Section 15.9 for calculation of breakdown.

12.1.3 Analysis of the DDT/endrin breakdown solution must show the breakdown of DDT and endrin to be less than 15% for each component.

12.2 Extracted sample batches

12.2.1 Extracted sample batches consist of a maximum of 20 samples, 1 MPB, 1 MS, 1 MSD, LCS, and 1 LCSD.

12.2.2 All 20 samples and the QC samples are spiked with known surrogate concentrations. The recoveries for these compounds are used as indicators of possible problems that may occur during extraction of individual samples.

12.2.3 The MS, MSD, LCS, and LCSD are spiked at a known concentration with either pesticides or PCBs.

12.3 Initial CALs

12.3.1 A set of five or more calibration levels (Section 10.10.1.1) is analyzed for pesticides. (Refer to Section 15 for calculations and to Section 18.2 for acceptance criteria.) A minimum of five standard levels is used to determine the calibration of a linear curve. A minimum of six levels is used to determine the calibration of a quadratic curve.

12.3.2 Multiple component analytes (PCBs and toxaphene) use five or more CAL levels for the mixture of Aroclors 1016 and 1260 (Section 10.10.1.2) and for toxaphene (Section 10.10.1.3). A single point calibration WS is used for other Aroclors (Section 10.10.4) unless it is known that a certain Aroclor is present. In this situation, a multiple-point calibration curve may be analyzed.

12.3.3 Samples may be analyzed within the 12-hour time frame following completion of the initial calibration and providing that the ICV passes.

12.4 CCS – A check of the calibration curve must be performed once every 12 hours and every 20 samples or less. It is not necessary to analyze a CCS within the 12-hour time period in which the initial calibration was performed. The check must include at least two standards, one for pesticides and one for PCBs (Aroclors 1016 and 1260). If the criteria in Section 18.3.1 are met, the initial calibration curves are still valid.

12.5 Blanks – A SB or MPB must be analyzed before or after the CCS. A blank must be analyzed every 12 hours to show that the analytical system is free from interferences and contamination. A blank will be run more frequently if contamination is a concern. If the criteria in Section 18.6 or in Section 18.7 are met, the analysis of samples can begin.

12.6 Replicates

12.6.1 The laboratory analyzes a MS/MSD or a LCS/LCSD pair at a minimum of once every extraction batch to assess precision and accuracy.

12.6.2 If the sample associated with the MS and MSD does not meet surrogate acceptance criteria, Section 19.1.4 should be followed.

12.7 LCS/LCSD

12.7.1 One LCS must be prepared for every batch of samples. A LCSD must be prepared if a MS/MSD is not available, and should be prepared and included in the batch if the MS/MSD appear to be samples which could be problematic for the ASE or GPC.

12.7.2 Refer to Section 18.4 for acceptance criteria.

12.8 RT windows

12.8.1 Three sets of standards are analyzed over a 72-hour period. The standard deviation (SD) is determined and the RT window is the larger of 3X the SD or +/- 0.03 min. from the RT of a mid-point initial CAL.

12.8.2 Drifting of the surrogate compound RTs is used as an indicator of possible matrix induced drifting. Oil samples tend to have drifting RTs. The analyst's experience is depended upon in this situation.

12.9 ICV – The ICV is analyzed after every initial calibration and at least once per quarter. The ICV may be analyzed in place of the CCS. Refer to Section 18.3 for acceptance criteria.

12.10 Initial demonstration of acceptability - Each analyst must demonstrate initial proficiency with this method by generating data of acceptable accuracy and precision for the target analytes. This is accomplished by preparing and analyzing four aliquots of a reference sample for each scan.

12.10.1 Reference sample preparation - Five aliquots of reagent water or sand are extracted using SOP 401 or SOP 405. One of these aliquots is a MPB. The other four are spiked at a mid-range concentration with all of the pesticide single compound analytes listed in Section 4.1. This is repeated for the Aroclor mix of 1016 and 1260 and for toxaphene.

12.10.2 The normal sample cleanup methods are used for sample extracts.

12.10.3 Calculate the recovery of each compound (Section 15.5). Compare to the laboratory generated limits.

12.10.4 Calculate the SD of each compound and compare them to laboratory generated limits.

12.11 Surrogate compounds are added to all samples, MPBs, and QC samples during extraction. Surrogate recovery data is compared to DEQ Laboratory generated limits.

13.0 Calibration and Standardization

13.1 Initial calibration curve - The GC system is calibrated prior to the analysis of samples. The WSs prepared in Section 10.10.1 are analyzed. A minimum of five levels are used for each calibration curve for pesticides and toxaphene and for the mixture of Aroclors 1016 and 1260. Single-point calibrations are normally used for the rest of the Aroclors. A mixture of Aroclors 1016 and 1260 will include many of the peaks found in the other Aroclors. A calibration of 1016/1260 with 5 concentration levels should be sufficient to determine the linearity of the detector response. Single-point standards at the linear range midpoint of the detector for the other Aroclors can be used to determine the calibration factor of those Aroclors. All standards, blanks, samples, and QC are injected at the same volume (usually 2 µL). The initial calibration is performed whenever the CCS fails to meet acceptance criteria or when major instrument repairs are made.

13.1.1 Evaluate the initial calibration (Section 18.2). If the criteria are met, analysis of the ICV may proceed. If the criteria are not met, evaluate the calibration using the linear curve or the quadratic curve or perform instrument maintenance and repeat.

13.2 ICV - The ICV is analyzed after every acceptable initial calibration to verify the accuracy of the initial CALs. The ICV is prepared as described in Section 10.10.2.

13.2.1 Calculate the recovery of each analyte (Section 15.5).
13.2.2 Evaluate the ICV (Section 18.3). If the ICV meets the criteria, proceed with Section 14. If the ICV does not meet the criteria, maintenance should be performed as needed.

13.3 SB - A SB is analyzed after every calibration curve to ensure that the system is contamination free.

13.3.1 Place 1 mL of hexane into a crimp cap vial. Cap. Inject onto the GC.
13.3.2 Evaluate the data (Section 18.6).

14.0 Procedure

14.1 Blank – A MPB or a SB is analyzed once every 12-hour shift in which samples are analyzed.

14.1.1 Evaluate the data (Section 18.6 or Section 18.7).

14.2 CCS - The CCS verifies that the initial calibration curve is still valid. It is analyzed once every 12-hour shift and every 20 samples or less. The concentration may alternate between a high standard and a low standard. The concentration is prepared as described in Section 10.10.1.

14.2.1 Calculate the % difference from the initial calibration (Section 15.7).
14.2.2 Evaluate the data (Section 18.3). If the criteria are met, proceed with Section 14.3. If the criteria are not met, maintenance should be performed as needed, or a new calibration should be performed.

14.3 Sample analysis

14.3.1 Load the samples onto the autosampler. (See Attachment 22.5 for an example). Analyze.
14.3.2 Evaluate each target compound (Section 18.1) and chromatogram (Section 18.5).
14.3.3 Calculate target analyte concentrations (Section 15.1 and/or Section 15.2).
14.3.4 Calculate the recovery of each spiked compound (Section 15.4 and Section 15.5).
14.3.5 Calculate the relative percent difference (RPD) of the MS/MSD or LCS/LCSD pair (Section 15.6).
14.3.6 Evaluate the recovery and RPD (Section 18.4).

14.4 Preventative maintenance

14.4.1 The septa, insert, and gold seal are changed as needed.
14.4.2 The injection port is cleaned as needed.
14.4.3 The injection end of the capillary column is clipped as needed.
14.4.4 The autosampler syringe is changed as needed.

15.0 Calculations

15.1 Single peak target analytes – Identified target analytes should be calculated by the external calibration method.

15.1.1 Aqueous samples

$$\text{concentration (ug/L)} = (A_{ini})\frac{(Df)(M)(Vt)}{(V)}$$

A_{ini} = initial analyte concentration (ug/L).

"Raw Amount" of TC Workstation is the initial analyte concentration (A_{ini}). (For multiple component analytes, the total Raw Amount is an average of the individual peak Raw Amounts.)

Df = dilution factor.
M = multiplier (usually 2.0 from GPC cleanup).
Vt = total volume of final extract after florisil (usually 1 mL).
V = volume of sample extracted (mL).

15.1.2 Solid samples

15.1.2.1 Pest/PCB (not PCB(DRO) extracted samples)

$$\text{concentration (ug/Kg)} = (A_{ini})\frac{(Df)(M)(Vt)}{(W)(\%S)}$$

A_{ini} = initial analyte concentration (ug/L).

"Raw Amount" of TC Workstation is the initial analyte concentration (A_{ini}). *(For multiple component analytes, the total Raw Amount is an average of the individual peak Raw Amounts.)*

Df = dilution factor.
M = multiplier (usually 2.0 from GPC cleanup).
Vt = total volume after ASE extraction (usually 5 mL).
W = weight of sample extracted (usually 10 grams).
%S = %solids/100.

15.1.2.2 Soil Samples (PCB(DRO) extracted samples)

$$\text{concentration (ug/Kg)} = \frac{(A_{ini})(Df)(M)(Vt)}{(W)(\%S)}$$

A_{ini} = initial analyte concentration (ug/L).

"Raw Amount" of TC Workstation is the initial analyte concentration (A_{ini}). *(For PCBs, the total Raw Amount is an average of the individual peak Raw Amounts.)*

Df = dilution factor.
M = multiplier (usually 2.0 from GPC cleanup).
Vt = total volume after ASE extraction (usually 10 mL).
W = weight of sample extracted (usually 25 grams).
%S= % solids/100.

15.1.3 Oil or liquid waste samples

$$\text{concentration (mg/Kg)} = \frac{(A_{ini})(Df)(M)(Vt)(SG)}{(W)(1000)}$$

A_{ini} = initial analyte concentration (ug/L).

"Raw Amount" of TC Workstation is the initial analyte concentration (A_{ini}). *(For PCBs, the total Raw Amount is an average of the individual peak Raw Amounts.)*

Df = dilution factor.
M = multiplier (usually 2.0 from GPC cleanup).
Vt = total volume after waste dilution (usually 10 mL).
SG=Silica gel factor (usually 0.5 for oils).
W = weight of sample extracted (usually 0.5 grams).
1000=conversion factor to mg/Kg.

Note: Oil samples are usually diluted 0.5 g to 10 mL. If GPC is performed, 2 mL of 10 are diluted again to 10 mL by the GPC unit. Five mL of this are actually injected onto the GPC column. The final extract is concentrated to 1 mL after silica gel clean up.

15.2 Multiple peak target compound calculations**.**

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15.2.1 The analyst must be experienced in recognizing the peak pattern of the compounds prior to calculation.

15.2.2 A number of major peaks (usually ten) are selected from the standard and calibrated. If no interferences are apparent, a minimum of three of these peaks is used for quantitation. The average concentration of the usable peaks represents the concentration of the Aroclor in the sample. For questionable patterns, the closest representative Aroclors are quantitated, and a % relative standard deviation (RSD) of the peaks used is figured. Analyst experience and the %RSD will determine the specific Aroclor calibration to be used for quantitation. If multiple Aroclors are present, the analyst chooses peaks from each Aroclor that are not significant peaks in the other Aroclors. At least three peaks should be used for quantitation of each Aroclor. Analyst experience is important in recognition and reporting of multiple Aroclors. Use section 15.1 for calculations.

15.3 Surrogate compound recovery - Calculate the concentration of each surrogate compound (Section 15.1). Calculate the recovery of each surrogate compound in all samples, blanks, MSs, MSDs, LCSs, LCSDs, CCSs, and ICVs as follows:

$$\%\text{recovery} = \frac{\text{concentration found} \times 100}{\text{concentration spiked}}$$

15.4 MS/MSD %recovery - Calculate the concentration of each MS or MSD analyte (Section 15.1). Calculate the recovery of each MS or MSD analyte as follows:

$$\text{MS or MSD \%recovery} = \frac{(SSR - SR) \times 100}{SA}$$

SSR = analyte concentration in spike.
SR = analyte concentration in unspiked sample.
SA = spike added.

NOTE: Make sure that all units are the same.

15.5 LCS, LCSD, CCS, reference sample, and ICV %recovery - Calculate the concentration of each analyte (Section 15.1). Calculate the recovery of each analyte as follows:

$$\%\text{recovery} = \frac{SSR \times 100}{SA}$$

SSR = analyte concentration in spike.
SA = spike added.

NOTE: Make sure that all units are the same.

15.6 Relative Percent Difference (RPD) - Calculate the RPD of the recoveries of each compound in the MS/MSD or LCS/LCSD as follows:

$$RPD = \frac{(MS - MSD) \times 100}{} \quad \text{or} \quad RPD = \frac{(LCS - LCSD) \times 100}{}$$

(1/2)(MS + MSD) (1/2)(LCS + LCSD)

MS = matrix spike recovery.
MSD = matrix spike duplicate recovery.
LCS = laboratory control sample recovery.
LCSD = laboratory control samples duplicate recovery.

NOTE: The formula above is the absolute value of the difference. RPD is always expressed as a positive value.

15.7 Percent difference

$$\% \text{ difference} = \frac{(\text{Concentration of CAL} - \text{Concentration of CCS}) \times 100}{\text{Concentration of CAL}}$$

15.8 Least squares regression for linear and quadratic calibrations.

15.8.1 The equation is as follows when using a linear regression of the peak area response (y) versus the concentration of the standards (x):

$y = ax + b$.
a = slope.
b = intercept.

15.8.2 The equation is as follows when using a quadratic fit:

$Y= c_0 +c_1x+c_2x^2$ where the c's are the curve coefficients in the method

NOTE: This line should not be forced through the origin, should include at least five points for linear calibrations, should include at least six points for quadratic calibrations, and should not include a blank.

15.9 DDT/endrin breakdown calculation – Use area counts in all calculations.

$$\text{DDT breakdown} = \frac{(\text{DDE} + \text{DDD})}{(\text{DDE} + \text{DDD} + \text{DDT})} \times 100$$

$$\text{Endrin breakdown} \frac{(\text{EA} + \text{EK})}{(\text{EA} + \text{EK} + \text{E})} \times 100$$

EA = endrin aldehyde.
EK = endrin ketone.
E = endrin.

16.0 Method Performance

16.1 Each MDL must be labeled with: the instrument number, the analyst's name(s), the dates of extraction and analysis, the GPC number, the ASE number, and the extraction technician's name(s).

17.0 Pollution Prevention

17.1 This method uses small quantities of organic solvents. The waste that is produced is collected for disposal as chlorinated waste.

17.2 For information concerning pollution prevention that may be applicable to laboratory operations, consult Less is Better: Laboratory Chemical Management for Waste Reduction available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW, Washington, DC 20036.

18.0 Data Assessment

18.1 Qualitative analysis - All target analytes are identified by comparing the RT of that component to the RT of that compound in the standard on both columns (Attachment 22.9).

18.1.1 RT - The RT must be within the established RT window of the standard peak.

18.1.2 Multiple component compounds (Aroclors and toxaphene) are analyzed on one column. The pattern of the compound is compared to a standard on that column. Both RT and pattern must match for confirmation of these analytes (Attachment 22.8).

18.2 Initial calibration evaluation - The correlation coefficient for the linear calibration using a least squares regression must be 0.99 or greater. The coefficient of determination for a quadratic calibration must be 0.99 or greater. For a linear calibration using the average calibration factor, the RSD has to be less than or equal to 20% to assume linearity through the origin. If a reporting level standard does not quantitate an appropriate number with a linear calibration, the quadratic calibration should be used.

18.3 ICV and CCS evaluation

18.3.1 ICV/CCS - Individual analytes that exceed the 15% limit must be coded as estimated (Section 15.7) in samples or reanalyzed.

18.4 MS, MSD, LCS, and LCSD evaluation - Recoveries for each spiked analyte are calculated as in Section 15 and must meet DEQ Laboratory generated limits, or appropriate coding is necessary.

18.5 Target analyte evaluation

18.5.1 Responses and RTs in all standards should be evaluated during or immediately after data acquisition. If the RTs change by more than 30 seconds from the most recent CCS (12-hour), the chromatographic system must be inspected for malfunctions and corrections made as required. For samples analyzed during the same 12-hour time period as the initial CALs, compare the responses and RTs against the mid-range CAL. If target analytes are reported below the RL, they must meet the identification

criteria and results estimated (J code). Aroclors and DDT, DDD, and DDE are usually the only analytes reported below the RL.

18.5.2 Multiple peak target compounds are usually analyzed on the primary column (ZB-5 or equivalent). When a PCB or toxaphene is suspected, the PCB or toxaphene pattern and the RTs of individual peaks are compared to a standard on that column. Correct RTs and similar patterns must be present to confirm the multiple peak analytes. A second column pattern match may also be used as additional confirmation.

18.6 SB evaluation – The SB must not contain target analytes above the RL.

18.7 MPB evaluation – The method blank should not contain target analytes above the RL. A note reporting the level found in the MPB must accompany any positive hits in samples if these analytes are found.

18.8 Data review – All data is reviewed by a second person prior to release.

18.9 Surrogate evaluation – Recoveries for each surrogate compound are calculated as in section 15.3. They must meet DEQ Laboratory generated limits, or corrective action must be taken (Section 19.1). Dilutions of 1:10 or greater dilute the surrogate recovery. A value may not be reported in this situation.

19.0 Corrective Actions

19.1 Surrogate recovery - All surrogate recoveries must fall within the specified laboratory generated windows. If any surrogate compound is out of control due to obvious matrix problems, the sample is not re-run, and the sample result(s) will be qualified as estimated. If the sample has a surrogate that is out of control for no apparent reason, the sample will be re-extracted if additional sample is available. If the reanalysis is within limits, the first analysis will be discarded, and the second analysis reported. If the second analysis agrees with the first, the poor recovery is attributed to matrix interference. Results and RLs from the first analysis will be reported with the appropriate qualifier. If numerous samples are out of control for no apparent reason, all instrumentation used in extraction, cleanup, and analysis should be inspected for malfunctions. Corrections must be made as needed. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary. If the recovery is outside of acceptance limits for samples, any values obtained from any positively identified compounds must be reported as estimated (J code). If the recovery of any one surrogate is not within limits, the following are required:

19.1.1 Check all calculations and instrument performance. Check the preparation of the surrogate spiking solution for errors or degradation.

19.1.2 Reanalyze the sample unless a calculation error was found.

19.1.3 If an undiluted analysis with acceptable surrogate recoveries is being submitted, do not reanalyze diluted samples if the surrogate recoveries are outside the limits.

19.1.4 If the sample associated with the MS/MSD does not meet the criteria, it should be reanalyzed only if the MS/MSD surrogate recoveries are within the limits. If the

sample and associated MS/MSD show the same pattern (i.e., outside the limits), the sample does not require reanalysis.

19.1.5 If surrogate recoveries still fall outside the acceptance limits and if additional sample is available, re-extraction and reanalysis should be performed. If the results are the same, the first analysis may be used. If results are not the same and if the second extraction is out of hold time, the sample must be coded H.

19.2 MS/MSD/LCS/LCSD recoveries - If any analyte percent recovery in the MS or in the MSD falls outside the acceptance windows, the laboratory must determine if there is a matrix effect or a laboratory performance problem. A matrix effect is indicated if the LCS data are within limits but the MS or MSD data exceeds the limits. If a matrix effect is determined, the report will be flagged as matrix interference. If recoveries of the analytes in the LCS are outside acceptance limits, the problem may be with the extraction or analytical system. Once the problem has been resolved, corrective action may include reextraction and/or reanalysis of samples.

19.3 RPD - If the RPD of any analyte is outside the acceptance range, the system should be evaluated in order to locate potential problems. Once the problem has been resolved, corrective action may include reextraction and/or reanalysis of samples, or the data may be flagged as estimated.

19.4 SB – SB results must be less than the RL for the target analyte. If the SB does not meet this criterion, action should be taken to locate and correct the source of the contamination prior to the analysis of samples, QA/QC samples, etc.

19.5 MPB – MPB results should be less than the RL for each analyte. If the MPB does not meet this criterion, action should be taken to locate and reduce the source of the contamination. Samples that contain positive hits of analytes found in the MPB should be re-extracted and/or reanalyzed if possible. If the MPB results do not meet criteria and reanalysis is not possible, the MPB results must be reported.

19.6 Hold times - All analysis dates must be within 40 days of the extraction date. If analysis is past the hold time, all data must be flagged as estimated.

19.7 If the CCS is outside the 15% criteria, samples should be evaluated and reanalyzed if appropriate.

20.0 Waste Management

20.1 The DEQ Laboratory Safety Officer arranges for proper waste disposal. All waste should be put in the proper containers (i.e., glass in metal cans for glass disposal, etc.).

21.0 References

21.1 United States Code of Federal Regulations, Title 40, U.S. Government Printing Office, Washington, DC, Appendix A and B, Part 136.

21.2 Test Methods for Evaluating Solid Waste, U.S. Environmental Protection Agency, Document SW-846, Third Edition 1986, Final Update III, December1996.

21.3 HP 5890 Gas Chromatograph Manual.

21.4 Perkin Elmer Turbochrom Workstation Software Manual.

21.5 HP 6890 Gas Chromatograph Manual.

21.6 Analytical Chemistry of PCBs, 2nd Edition, by Mitchell D. Erikson, Lewis Publishers, N.Y., 1997

22.0 Attachments

22.1 Hexane MSDS.

22.2 25 component pesticide standard, part # 93238, Absolute Standards, 7 component VOC standard, part #93237, Absolute Standards.

22.3 Stock standard logbook page.

22.4 Semivolatile standard logbook page.

22.5 Pesticide/PCB run logbook page.

22.6 Target analyte RLs and units.

22.7 GC operating conditions.

22.8 Chromatogram comparisons.

22.9 RT differences – Channels A and B.

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Signature Page

APPROVED BY _____ DATE 2/28/2007
CHIEF, LABORATORY SERVICES SECTION

APPROVED BY _____ DATE 2/28/07
COMPLIANCE OFFICER

APPROVED BY _____ DATE 2-27-07
UNIT MANAGER

APPROVED BY _____ DATE 2/26/07
SECOND SHIFT MANAGER

APPROVED BY _____ DATE 2/27/07
ANALYST

Material Safety Data Sheet

n-Hexane

Section 1. Product and Company Identification

Product name : n-Hexane
Product code : HX0295
Synonym : Hexane
Material uses : Other non-specified industry: Analytical reagent.
Manufacturer : EMD Chemicals Inc.
P.O. Box 70
480 Democrat Road
Gibbstown, NJ 08027
856-423-6300 Technical Service
Monday - Friday: 8:00 - 5:00 PM
Validation date : **5/30/2006.**
Print date :
In case of emergency : 800-424-9300 CHEMTREC (USA)
613-996-6666 CANUTEC (Canada)
24 Hours/Day: 7 Days/Week

Section 2. Hazards Identification

Physical state : Liquid.
Odor : Characteristic.
OSHA/HCS status : This material is considered hazardous by the OSHA Hazard Communication Standard (29 CFR 1910.1200).
Emergency overview : DANGER!
EXTREMELY FLAMMABLE LIQUID AND VAPOR.
HARMFUL IF INHALED OR SWALLOWED.
CAUSES RESPIRATORY TRACT, EYE AND SKIN IRRITATION.
CAUSES DAMAGE TO THE FOLLOWING ORGANS: PERIPHERAL NERVOUS SYSTEM, RESPIRATORY TRACT, SKIN, CENTRAL NERVOUS SYSTEM, EYE, LENS OR CORNEA.
VAPOR MAY CAUSE FLASH FIRE.
ASPIRATION HAZARD.
Do not ingest. Avoid contact with skin and clothing. Avoid breathing vapor or mist. Keep away from heat, sparks and flame. Keep container closed. Use only with adequate ventilation. Wash thoroughly after handling.
Routes of entry : Dermal contact. Eye contact. Inhalation. Ingestion.
Potential acute health effects
Eyes : Irritating to eyes.
Skin : Irritating to skin.
Inhalation : Toxic by inhalation. Irritating to respiratory system.
Ingestion : Toxic if swallowed.
Carcinogenic effects : No known significant effects or critical hazards.
Mutagenic effects : No known significant effects or critical hazards.
Teratogenicity / Reproductive toxicity : No known significant effects or critical hazards.
Medical conditions aggravated by over-exposure : Repeated skin exposure can produce local skin destruction or dermatitis. Repeated or prolonged exposure to the substance can produce lung damage. Repeated or prolonged contact with spray or mist may produce chronic eye irritation and severe skin irritation. Repeated or prolonged exposure to the substance can produce target organs damage.

See toxicological information (section 11)

Section 3. Composition/Information on Ingredients

United States

Name	CAS number	% by Weight
n-Hexane	110-54-3	100

Hexane is primarily n-Hexane with Hexane isomers Methylcyclopentane, 2-Methylpentane, 3-Methylpentane.

Section 4. First Aid Measures

Eye contact : Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Check for and remove any contact lenses. Get medical attention.
Skin contact : Get medical attention immediately. Flush contaminated skin with plenty of water. Continue to rinse for at least 10 minutes. Remove contaminated clothing and shoes. Wash contaminated clothing thoroughly with water

before removing or wear gloves. Wash clothing before reuse. Clean shoes thoroughly before reuse.

Inhalation : Get medical attention immediately. Move exposed person to fresh air. If it is suspected that fumes are still present, the rescuer should wear an appropriate mask or self-contained breathing apparatus. Keep person warm and at rest. If not breathing, if breathing is irregular or if respiratory arrest occurs, provide artificial respiration or oxygen by trained personnel. It may be dangerous to the person providing aid to give mouth-to-mouth resuscitation. If unconscious, place in recovery position and get medical attention immediately. Maintain an open airway. Loosen tight clothing such as a collar, tie, belt or waistband.

Ingestion : Get medical attention immediately. Wash out mouth with water. Remove dentures if any. Move exposed person to fresh air. Keep person warm and at rest. If material has been swallowed and the exposed person is conscious, give small quantities of water to drink. Stop if the exposed person feels sick as vomiting may be dangerous. Do not induce vomiting unless directed to do so by medical personnel. If vomiting occurs, the head should be kept low so that vomit does not enter the lungs. Never give anything by mouth to an unconscious person. If unconscious, place in recovery position and get medical attention immediately. Maintain an open airway. Loosen tight clothing such as a collar, tie, belt or waistband.

Protection of first-aiders : No action shall be taken involving any personal risk or without suitable training. If it is suspected that fumes are still present, the rescuer should wear an appropriate mask or self-contained breathing apparatus. It may be dangerous to the person providing aid to give mouth-to-mouth resuscitation.

Section 5. Fire Fighting Measures

Flammability of the product : Extremely flammable liquid and vapor. Vapor may cause flash fire. Vapors may accumulate in low or confined areas or travel a considerable distance to a source of ignition and flash back. Runoff to sewer may create fire or explosion hazard.

Products of combustion : These products are carbon oxides (CO, CO_2).

Extinguishing media

Suitable : Use dry chemical, CO_2, water spray (fog) or foam.

Not suitable : Do not use water jet.

Special exposure hazards : Not available.

Special protective equipment for fire-fighters : Fire-fighters should wear appropriate protective equipment and self-contained breathing apparatus (SCBA) with a full face-piece operated in positive pressure mode.

Special remarks on fire hazards : Vapor may travel a considerable distance to source of ignition and flash back.

Section 6. Accidental Release Measures

Personal precautions : Immediately contact emergency personnel. Eliminate all ignition sources. Keep unnecessary personnel away. Use suitable protective equipment. Do not touch or walk through spilled material.

Environmental precautions : Avoid dispersal of spilled material and runoff and contact with soil, waterways, drains and sewers.

Methods for cleaning up : If emergency personnel are unavailable, contain spilled material. For small spills, add absorbent (soil may be used in the absence of other suitable materials) and use a non-sparking or explosion-proof means to transfer material to a sealable, appropriate container for disposal. For large spills, dike spilled material or otherwise contain material to ensure runoff does not reach a waterway. Place spilled material in an appropriate container for disposal.

Section 7. Handling and Storage

Handling : Do not ingest. Avoid contact with eyes, skin and clothing. Keep container closed. Use only with adequate ventilation. Avoid breathing vapor or mist. Keep away from heat, sparks and flame. To avoid fire or explosion, dissipate static electricity during transfer by grounding and bonding containers and equipment before transferring material. Use explosion-proof electrical (ventilating, lighting and material handling) equipment. Wash thoroughly after handling.

Storage : Store in a segregated and approved area. Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Avoid all possible sources of ignition (spark or flame).

Section 8. Exposure Controls/Personal Protection

Product name United States	Exposure limits
n-Hexane	**OSHA (United States, 1989).** TWA: 180 mg/m^3 **NIOSH REL (United States, 12/2001).** TWA: 180 mg/m^3 10 hour/hours. Form: All forms TWA: 50 ppm 10 hour/hours. Form: All forms **OSHA PEL (United States, 8/1997).** TWA: 1800 mg/m^3 8 hour/hours. Form: All forms TWA: 500 ppm 8 hour/hours. Form: All forms **OSHA PEL 1989 (United States, 3/1989).**

TWA: 180 mg/m^3 8 hour/hours. Form: All forms
TWA: 50 ppm 8 hour/hours. Form: All forms
ACGIH TLV (United States, 1/2006). Skin Notes: Substances for which there is a Biological Exposure Index or Indices 1998 Adoption.
TWA: 50 ppm 8 hour/hours. Form: All forms

Consult local authorities for acceptable exposure limits.

Engineering measures : Use only with adequate ventilation. If user operations generate dust, fumes, vapor or mist, use process enclosures, local exhaust ventilation or other engineering controls to keep worker exposure to airborne contaminants below any recommended or statutory limits. The engineering controls also need to keep gas, vapor or dust concentrations below any lower explosive limits. Use explosion-proof ventilation equipment.

Personal protection

Eyes : Safety eyewear complying with an approved standard should be used when a risk assessment indicates this is necessary to avoid exposure to liquid splashes, mists, gases or dusts.
Recommended: splash goggles

Skin : Personal protective equipment for the body should be selected based on the task being performed and the risks involved and should be approved by a specialist before handling this product.
Body: Recommended: lab coat

Respiratory : Use a properly fitted, air-purifying or air-fed respirator complying with an approved standard if a risk assessment indicates this is necessary. Respirator selection must be based on known or anticipated exposure levels, the hazards of the product and the safe working limits of the selected respirator.

Hands : Chemical-resistant, impervious gloves complying with an approved standard should be worn at all times when handling chemical products if a risk assessment indicates this is necessary. Recommended: nitrile rubber

Hygiene measures : Wash hands, forearms and face thoroughly after handling chemical products, before eating, smoking and using the lavatory and at the end of the working period. Appropriate techniques should be used to remove potentially contaminated clothing. Wash contaminated clothing before reusing. Ensure that eyewash stations and safety showers are close to the workstation location.

Section 9. Physical and Chemical Properties

Physical state : Liquid.
Flash point : Closed cup: -23.15°C (-9.7°F).
Auto-ignition temperature : 224.85°C (436.7°F)
Flammable limits : Lower: 1.1% Upper: 7.5%
Color : Colorless.
Odor : Characteristic.
Molecular weight : 86.2 g/mole
Molecular formula : C6-H14
Boiling/condensation point : 68.89°C (156°F)
Melting/freezing point : -139.44°C (-219°F)
Critical temperature : 234.3°C (453.7°F)
Relative density : 0.659 (Water = 1)
Vapor density : 3 (Air = 1)
Odor threshold : 130 ppm
Evaporation rate : 6.82 compared with Butyl acetate.

Section 10. Stability and Reactivity

Stability and reactivity : The product is stable.
Incompatibility with various substances : Highly reactive or incompatible with the following materials: oxidizing materials.
Hazardous decomposition products : carbon oxides (CO, CO_2)
Hazardous polymerization : Will not occur.
Conditions of reactivity : Extremely flammable in the presence of the following materials or conditions: open flames, sparks and static discharge, heat, shocks and mechanical impacts and oxidizing materials.
Vapor may travel a considerable distance to source of ignition and flash back.
Extremely explosive in the presence of the following materials or conditions: heat, shocks and mechanical impacts and oxidizing materials.
Highly explosive in the presence of the following materials or conditions: open flames, sparks and static discharge.

Section 11. Toxicological Information

Toxicity data
United States

Product/ingredient name	Test	Result	Route	Species
n-Hexane	LD50	25000 mg/kg	Oral	Rat
	LC50	48000 ppm (4 hour/hours)	Inhalation	Rat

Chronic effects on humans : Causes damage to the following organs: peripheral nervous system, upper respiratory tract, skin, central nervous system (CNS), eye, lens or cornea.

Other toxic effects on humans : Very hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation (lung irritant).

Specific effects

Carcinogenic effects : No known significant effects or critical hazards.

Mutagenic effects : No known significant effects or critical hazards.

Teratogenicity / Reproductive toxicity : No known significant effects or critical hazards.

Sensitization

Ingestion : No known significant effects or critical hazards.

Inhalation : Irritating to respiratory system.

Eyes : Irritating to eyes.

Skin : Irritating to skin.

Section 12. Ecological Information

Ecotoxicity data

United States

Product/ingredient name	Species	Period	Result
n-Hexane	Pimephales promelas (LC50)	96 hour/hours	2.5 mg/l

Environmental precautions : Toxic to aquatic organisms. May cause long-term adverse effects in the aquatic environment.

Products of degradation : These products are carbon oxides (CO, CO_2) and water.

Toxicity of the products of biodegradation : The products of degradation are less toxic than the product itself.

Section 13. Disposal Considerations

Waste disposal : The generation of waste should be avoided or minimized wherever possible. Avoid dispersal of spilled material and runoff and contact with soil, waterways, drains and sewers. Disposal of this product, solutions and any by-products should at all times comply with the requirements of environmental protection and waste disposal legislation and any regional local authority requirements.

Disposal should be in accordance with applicable regional, national and local laws and regulations. Local regulations may be more stringent than regional or national requirements.

The information presented below only applies to the material as supplied. The identification based on characteristic(s) or listing may not apply if the material has been used or otherwise contaminated. It is the responsibility of the waste generator to determine the toxicity and physical properties of the material generated to determine the proper waste identification and disposal methods in compliance with applicable regulations.

Refer to Section 7: HANDLING AND STORAGE and Section 8: EXPOSURE CONTROLS/PERSONAL PROTECTION for additional handling information and protection of employees.

Section 14. Transport Information

Regulatory information	UN number	Proper shipping name	Class	PG*	Label	Additional information
DOT Classification	UN1208	HEXANE	3	II		**Reportable quantity** 5000 lbs. (2268 kg)

PG* : Packing group

Section 15. Regulatory Information

United States

HCS Classification : Flammable liquid
Toxic material
Irritating material
Target organ effects

U.S. Federal regulations : TSCA 8(b) inventory: Listed
SARA 302/304/311/312 extremely hazardous substances: No products were found.
SARA 302/304 emergency planning and notification: No products were found.
SARA 302/304/311/312 hazardous chemicals: n-Hexane
SARA 311/312 MSDS distribution - chemical inventory - hazard identification: n-Hexane : Fire hazard, Immediate (acute) health hazard, Delayed (chronic) health hazard
Clean Water Act (CWA) 307: No products were found.
Clean Water Act (CWA) 311: No products were found.
Clean Air Act (CAA) 112 accidental release prevention: No products were found.
Clean Air Act (CAA) 112 regulated flammable substances: No products were found.
Clean Air Act (CAA) 112 regulated toxic substances: No products were found.

SARA 313

Product name	CAS number	Concentration

Form R - Reporting requirements :

n-Hexane	110-54-3	100

Supplier notification :

n-Hexane	110-54-3	100

SARA 313 notifications must not be detached from the MSDS and any copying and redistribution of the MSDS shall include copying and redistribution of the notice attached to copies of the MSDS subsequently redistributed.

State regulations : Pennsylvania RTK: n-Hexane : (generic environmental hazard)
Massachusetts RTK: n-Hexane
New Jersey: n-Hexane

Canada

WHMIS (Canada) : Class B-2: Flammable liquid
Class D-2B: Material causing other toxic effects (Toxic).

CEPA DSL/CEPA NDSL : CEPA DSL: n-Hexane

This product has been classified according to the hazard criteria of the CPR and the MSDS contains all the information required by the CPR.

EU regulations

Hazard symbol/symbols :

Risk phrases : R11- Highly flammable.
R62- Possible risk of impaired fertility.
R38- Irritating to skin.
R67- Vapors may cause drowsiness and dizziness.
R51/53- Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.

Safety phrases : S2- Keep out of the reach of children.
S9- Keep container in a well-ventilated place.
S16- Keep away from sources of ignition - No smoking.
S29- Do not empty into drains.
S33- Take precautionary measures against static discharges.
S36/37- Wear suitable protective clothing and gloves.
S61- Avoid release to the environment. Refer to special instructions/Safety data sheets.
S62- If swallowed, do not induce vomiting: seek medical advice immediately and show this container or label.

International regulations

International lists : Australia (NICNAS): n-Hexane

China: n-Hexane

Germany water class: n-Hexane

Japan (METI): n-Hexane

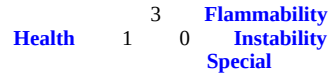
Korea (TCCL): n-Hexane

Philippines (RA6969): n-Hexane

Section 16. Other Information

Label requirements : DANGER!
EXTREMELY FLAMMABLE LIQUID AND VAPOR.
HARMFUL IF INHALED OR SWALLOWED.
CAUSES RESPIRATORY TRACT, EYE AND SKIN IRRITATION.
CAUSES DAMAGE TO THE FOLLOWING ORGANS: PERIPHERAL NERVOUS SYSTEM, RESPIRATORY TRACT, SKIN, CENTRAL NERVOUS SYSTEM, EYE, LENS OR CORNEA.
VAPOR MAY CAUSE FLASH FIRE.
ASPIRATION HAZARD.

National Fire Protection Association (U.S.A.) :



Notice to reader

The statements contained herein are based upon technical data that EMD Chemicals Inc. believes to be reliable, are offered for information purposes only and as a guide to the appropriate precautionary and emergency handling of the material by a properly trained person having the necessary technical skills. Users should consider these data only as a supplement to other information gathered by them and must make independent determinations of suitability and completeness of information from all sources to assure proper use, storage and disposal of these materials and the safety and health of employees and customers and the protection of the environment. EMD CHEMICALS INC. MAKES NO REPRESENTATION OR WARRANTY OF ANY KIND, EXPRESS OR IMPLIED, INCLUDING MERCHANTABILITY OR FITNESS FOR A PARTICULAR USE, WITH RESPECT TO THE INFORMATION HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS.

EC 2343 1,2 rec'd 4/10/

ABSOLUTE STANDARDS, INC • 44 Rossotto Drive • Hamden, CT • 06514 • (800)-368-1131

ISO 9001 Quality System Registered
DLS ANSI-RAB Accredited

CERTIFIED WEIGHT REPORT: Uncertainty < +,-0.5% Stated Values.

Part Number: 93238
Lot Number: 071602
Description: Pesticide Standard
25 components
Expiration Date: 071607

Solvent(s): Lot#
V43E34 Hexane (50%)
V14417 Toluene (50%)

Nominal Concentration(ug/mL): 1000

Weight(s) & Volume(s) shown below were combined and diluted to 10 mL

Formulated By:
Vincent K. Criscio, Jr. 071602 DATE

Reviewed By:
Pedro L. Rentas 071602 DATE

Compound	(RM#) Part Number	Lot Number	Dil. Factor	Initial Vol. (mL)	Initial Conc.(ug/mL)	Nominal Conc (µg/mL)	Purity (%)	Max. Target Weight(g)	*Actual Weight(g)	*Actual Conc (µg/mL)	MSDS Information (Solvent Safety Info. On Attached pg.) CAS#	OSHA PEL (TWA)	LD50
1. 2,2',4,4',5,5'-Hexabromobiphenyl	(1173)	NT01257	na	na	na	1000	97	0.01031	0.01032	1001.0	59080-40-9	N/A	N/A
2. a-Chlordane	93240	071602	1.00	10.00	1000.9	1000	na	na	na	1000.9	05103-71-9	0.5mg/m3 (skin)	orl-rat 500mg/kg
3. g-Chlordane	93240	071602	1.00	10.00	1001.5	1000	na	na	na	1001.5	05103-74-2	0.5mg/m3 (skin)	orl-rat 500mg/kg
4. Decachlorobiphenyl (209)	93240	071602	1.00	10.00	1001.5	1000	na	na	na	1001.5	02051-24-3	N/A	N/A
5. Hexabromobenzene	93240	071602	1.00	10.00	1001.0	1000	na	na	na	1001.0	00087-82-1	N/A	N/A
6. Mirex	93240	071602	1.00	10.00	1001.7	1000	na	na	na	1001.7	02385-85-5	N/A	orl-rat 306mg/kg
7. 2,4,5,6-Tetrachloro-m-xylene	93240	071602	1.00	10.00	1001.7	1000	na	na	na	1001.7	00877-09-8	N/A	N/A
8. Aldrin	93240	071602	1.00	10.00	1000.6	1000	na	na	na	1000.6	00309-00-2	0.25mg/m3 (skin)	orl-rat 39mg/kg
9. a-BHC	93240	071602	1.00	10.00	1000.6	1000	na	na	na	1000.6	00319-84-6	N/A	orl-rat 177mg/kg
10. b-BHC	93240	071602	1.00	10.00	1000.7	1000	na	na	na	1000.7	00319-85-7	N/A	orl-rat 6000mg/kg
11. d-BHC	93240	071602	1.00	10.00	1000.8	1000	na	na	na	1000.8	00319-86-8	N/A	orl-rat 1000mg/kg
12. g-BHC (Lindane)	93240	071602	1.00	10.00	1000.6	1000	na	na	na	1000.6	00058-89-9	0.5mg/m3 (skin)	orl-rat 76mg/kg
13. 4,4'-DDD	93240	071602	1.00	10.00	1000.5	1000	na	na	na	1000.5	00072-54-8	N/A	orl-rat 113mg/kg
14. 4,4'-DDE	93240	071602	1.00	10.00	1000.4	1000	na	na	na	1000.4	00072-55-9	N/A	orl-rat 880mg/kg
15. 4,4'-DDT	93240	071602	1.00	10.00	1000.9	1000	na	na	na	1000.9	00050-29-3	N/A	orl-rat 87mg/kg
16. Dieldrin	93240	071602	1.00	10.00	1000.6	1000	na	na	na	1000.6	00060-57-1	0.25mg/m3 (skin)	orl-rat 38300ug/kg
17. Endosulfan I	93240	071602	1.00	10.00	1000.5	1000	na	na	na	1000.5	00959-98-8	0.1mg/m3 (skin)	orl-rat 18mg/kg
18. Endosulfan II	93240	071602	1.00	10.00	1000.3	1000	na	na	na	1000.3	33213-65-9	0.1mg/m3 (skin)	orl-rat 18mg/kg
19. Endosulfan sulfate	93240	071602	1.00	10.00	1000.7	1000	na	na	na	1000.7	01031-07-8	N/A	orl-rat 18mg/kg
20. Endrin	93240	071602	1.00	10.00	1000.8	1000	na	na	na	1000.8	00072-20-8	0.1mg/m3 (skin)	orl-rat 7mg/kg
21. Endrin aldehyde	93240	071602	1.00	10.00	1000.8	1000	na	na	na	1000.8	07421-93-4	N/A	N/A
22. Endrin ketone	93240	071602	1.00	10.00	1000.8	1000	na	na	na	1000.8	53494-70-5	N/A	N/A
23. Heptachlor	93240	071602	1.00	10.00	1000.4	1000	na	na	na	1000.4	00076-44-8	0.5mg/m3 (skin)	orl-rat 40mg/kg
24. Heptachlor epoxide	93240	071602	1.00	10.00	1000.6	1000	na	na	na	1000.6	01024-57-3	N/A	orl-rat 47mg/kg
25. 4,4'-Methoxychlor	93240	071602	1.00	10.00	1000.6	1000	na	na	na	1000.6	00072-43-5	10mg/m3	orl-rat 6000mg/kg

EL 2241 1.5

Ridd 10/18/05

CERTIFIED WEIGHT REPORT

Part Number: 93237
Lot Number: 071602
Description: VOC Standard
7 components
Expiration Date: 071607

Nominal Concentration (µg/mL): 1000

Solvent(s): Lot # V34E62 Acetone

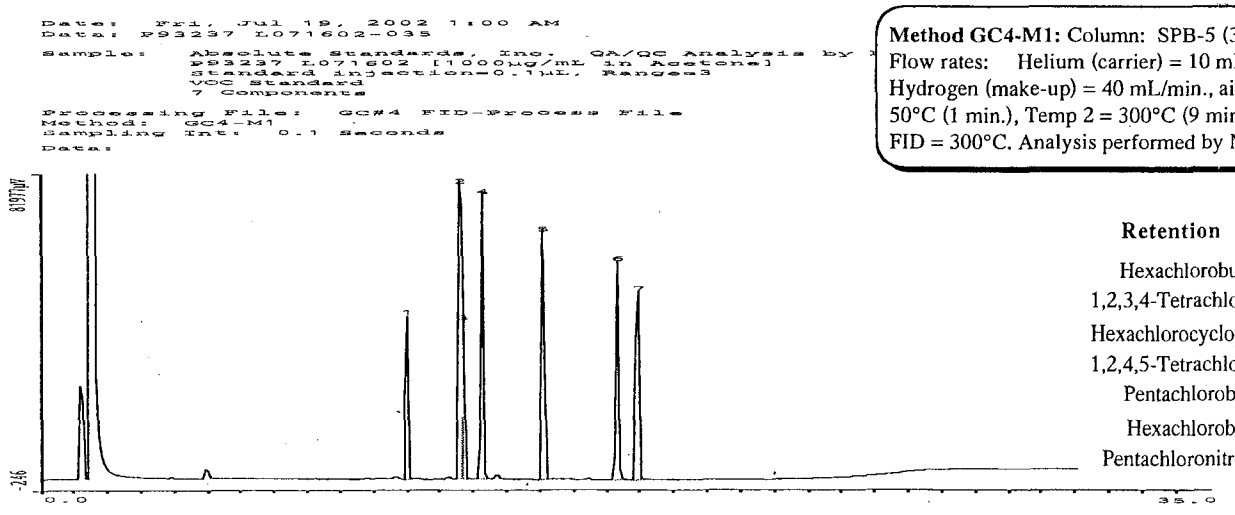
5E-05 Balance Uncertainty

Weight(s) shown below were combined and diluted to : 100.1 0.031 Flask Uncertainty

Lawrence Barry	071602
Formulated By: Lawrence Barry	DATE
Pedro L. Rentas	071602
Reviewed By: Pedro L. Rentas	DATE

MSDS Information
(Solvent Safety Info. On Attached pg.)

Compound	RM#	Lot Number	Nominal Conc (µg/mL)	Purity	Uncertainty: Purity	Target Weight(g)	Actual Weight(g)	*Actual Conc (µg/mL)	Expanded Uncertainty	CAS#	OSHA PEL (TWA)	LD50
1. Hexachlorobenzene	195	051697	1000	99	0.2	0.10107	0.10126	1001.9	0.0042	00118-74-1	N/A	orl-rat 10g/kg
2. Hexachlorobutadiene	197	08917HV	1000	98	0.2	0.10210	0.10240	1002.9	0.0042	00087-68-3	0.02 ppm (0.24mg/m3/8H)	orl-rat 90mg/kg
3. Hexachlorocyclopentadiene	198	07424DT	1000	98	0.2	0.10210	0.10233	1002.3	0.0042	00077-47-4	0.01 ppm (0.1mg/m3/8H)	orl-rat 1300mg/kg
4. Pentachlorobenzene	321	062627	1000	98	0.2	0.10210	0.10234	1002.4	0.0042	00608-93-5	N/A	orl-rat 1080mg/kg
5. Pentachloronitrobenzene (PCNB)	242	06505MW	1000	99	0.2	0.10107	0.10140	1003.3	0.0042	00082-68-8	N/A	orl-rat 1100mg/kg
6. 1,2,3,4-Tetrachlorobenzene	318	FBW01	1000	97.3	0.2	0.10283	0.10302	1001.8	0.0043	00634-66-2	N/A	orl-rat 1167mg/kg
7. 1,2,4,5-Tetrachlorobenzene	274	10408AS	1000	98	0.2	0.10210	0.10230	1002.0	0.0042	00095-94-3	N/A	orl-rat 1500mg/kg



Method GC4-M1: Column: SPB-5 (30m X 0.53mm X 1.5 µm film thickness), Flow rates: Helium (carrier) = 10 mL/min., Helium (make-up) = 15 mL/min., Hydrogen (make-up) = 40 mL/min., air (make-up) = 200 mL/min., Temp 1 = 50°C (1 min.), Temp 2 = 300°C (9 min.), Rate = 10°C/min., Injector= 200°C, FID = 300°C. Analysis performed by Nicole Davis.

Retention Time (min.)

Hexachlorobutadiene	11.00
1,2,3,4-Tetrachlorobenzene	12.61
Hexachlorocyclopentadiene	12.67
1,2,4,5-Tetrachlorobenzene	13.27
Pentachlorobenzene	15.08
Hexachlorobenzene	17.32
Pentachloronitrobenzene	17.91

P#93237 L#071602
Revision No.: 1, Date Issued 091599

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MDEQ-ENVIRONMENTAL LAB
STOCK TRACKING LOG

Environmental Lab Tracking Number	QTY (each)	Filley Tracking Number	Date Received (at lab)	Receiver Initials	Chemical,Analyte, Reagent or Solvent	Vendor and Catalog #	PO #	Manufacturer and Lot Number	Expiration Date
EL-									
EL-									
EL-									
EL-									
EL-									
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ATTACHMENT 22.3

FILE:ST-STD
12/30/98

MDEQ-ENVIRONMENTAL LAB
SEMI-VOLATILE ORGANIC STANDARDS

SVOC Tracking Number	Date Prepared	EL or SVOC Tracking Number	Analyte or Mix	Preparers Initials	Solvent	Solvent Tracking Number	Initial Concentration*	Initial Volume/Wt Standard*	Final Volume*	Final Concentration*	Expiration Date
SE-											
SE-											
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ATTACHMENT 22.4

FILE:SV-STD

*INCLUDE UNITS

Turbochrom Sequence File D:\TCWS Data\sequence\C-0624-2002.seq
Printed by : tcws40 on: 6/25/02 8:57:45 AM
Created by : tcws40 on: 6/24/02 2:19:10 PM
Edited by : tcws40 on: 6/25/02 8:57:19 AM
Number of Times Edited : 2
Description:

Sequence File Header Information:

Number of Rows : 119
Instrument Type : 900 Series Intelligent Interface
Injection Type : SINGLE

Sequence Sample Descriptions - Channel A

Row	Type	Name	Number	Study name	Sample Amt	Int Std Amt	Sample Vol	Dil Factor	Multiplier	Divisor
1	Sample	COLUMN PRIME	001	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
2	Sample	HEX	002	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
3	Sample	HEX	003	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
4	Sample	HEX	004	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
5	Sample	DDT/ENDRIN BREAKDOWN	005	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
6	Sample	AR 1016/1260 SE-665 25 PPB	006	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
7	Sample	AR 1016/1260 SE-664 50 PPB	007	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
8	Sample	AR 1016/1260 SE-663 100 PPB	008	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
9	Sample	AR 1016/1260 SE-662 200PPB	009	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
10	Sample	AR 1016/1260 SE-658 500 PPB	010	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
11	Sample	AR 1016/1260 SE-657 1000 PPB	011	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
12	Sample	AR 1016/1260 SE-656 2000 PPB	012	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
13	Sample	HEX	013	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
14	Sample	AR 1221/1262 500ppb SE-701	014	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
15	Sample	AR 1232/1268 500ppb SE-703	015	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
16	Sample	AR 1242 500ppb SE-566	016	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
17	Sample	AR 1248 500ppb SE-567	017	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
18	Sample	AR 1254 500ppb SE-568	018	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
19	Sample	PCB ICV 1660 500ppb SE-719	019	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
20	Sample	HEX	020	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
21	Sample	PEST 2.5 SE-739	021	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
22	Sample	PEST 5.0 SE-740	022	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
23	Sample	PEST 10 SE-741	023	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
24	Sample	PEST 30 SE-742	024	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
25	Sample	PEST 50 SE-743	025	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
26	Sample	PEST 125 SE-744	026	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
27	Sample	PEST 250 SE-738	027	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
28	Sample	ICV 100 PPB SE-680	028	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
29	Sample	PEST 10 SE-741	029	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
30	Sample	PEST 50 SE-743	030	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
31	Sample	AR 1016/1260 SE-664 50 PPB	031	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
32	Sample	AR 1016/1260 SE-658 500 PPB	032	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
33	Sample	TOX STD 500ppb SE-695	033	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
34	Sample	SYSTEM BLANK SE-1133	034	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
35	Sample	MPB 1530	035	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
36	Sample	LCS 1530	036	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
37	Sample	MS 1530	037	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
38	Sample	MSD 1530	038	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
39	Sample	06 184 01	039	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
40	Sample	06 184 02	040	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
41	Sample	06 184 03	041	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
42	Sample	06 184 04	042	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
43	Sample	06 184 05	043	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
44	Sample	AR 1016/1260 SE-664 50 PPB	044	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
45	Sample	AR 1016/1260 SE-658 500 PPB	045	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
46	Sample	PEST 10 SE-741	046	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
47	Sample	PEST 50 SE-743	047	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
48	Sample	SYSTEM BLANK SE-1133	048	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
49	Sample	06 184 06	049	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
50	Sample	06 184 07	050	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
51	Sample	06 184 08	051	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
52	Sample	06 184 09	052	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
53	Sample	06 184 10	053	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
54	Sample	06 184 11	054	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
55	Sample	06 184 12	055	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
56	Sample	06 165 01	056	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
57	Sample	06 132 01	057	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
58	Sample	HEX	058	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
59	Sample	AR 1016/1260 SE-664 50 PPB	059	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
60	Sample	AR 1016/1260 SE-658 500 PPB	060	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
61	Sample	PEST 10 SE-741	061	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
62	Sample	PEST 50 SE-743	062	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
63	Sample	SYSTEM BLANK SE-1133	063	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
64	Sample		064	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
65	Sample		065	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
66	Sample		066	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
67	Sample		067	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
68	Sample		068	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
69	Sample		069	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
70	Sample		070	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
71	Sample		071	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
72	Sample		072	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000

6/25/02 8:57:45 AM Sequence: D:\TCWS Data\sequence\C-0624-2002.seq

Sequence Sample Descriptions - Channel A

Row	Type	Name	Number	Study name	Sample Amt	Int Std Amt	Sample Vol	Dil Factor	Multiplier	Divisor
73	Sample		073	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
74	Sample		074	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
75	Sample		075	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
76	Sample		076	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
77	Sample		077	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
78	Sample		078	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
79	Sample		079	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
80	Sample		080	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
81	Sample		081	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
82	Sample		082	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
83	Sample		083	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
84	Sample		084	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
85	Sample		085	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
86	Sample		086	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
87	Sample		087	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
88	Sample		088	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
89	Sample		089	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
90	Sample		090	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
91	Sample		091	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
92	Sample		092	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
93	Sample		093	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
94	Sample		094	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
95	Sample		095	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
96	Sample		096	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
97	Sample		097	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
98	Sample		098	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
99	Sample		099	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
100	Sample		100	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
101	Sample		101	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
102	Sample		102	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
103	Sample		103	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
104	Sample		104	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
105	Sample		105	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
106	Sample		106	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
107	Sample		107	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
108	Sample		108	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
109	Sample		109	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
110	Sample		110	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
111	Sample		111	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
112	Sample		112	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
113	Sample		113	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
114	Sample		114	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
115	Sample		115	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
116	Sample		116	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
117	Sample		117	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
118	Sample		118	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000
119	Sample		119	PEST/PCB	1.000000	1.000000	1.000	1.000000	1.000000	1.000000

Row	Addend	Norm Factor
1	0.000000	100.000
2	0.000000	100.000
3	0.000000	100.000
4	0.000000	100.000
5	0.000000	100.000
6	0.000000	100.000
7	0.000000	100.000
8	0.000000	100.000
9	0.000000	100.000
10	0.000000	100.000
11	0.000000	100.000
12	0.000000	100.000
13	0.000000	100.000
14	0.000000	100.000
15	0.000000	100.000
16	0.000000	100.000
17	0.000000	100.000
18	0.000000	100.000
19	0.000000	100.000
20	0.000000	100.000
21	0.000000	100.000
22	0.000000	100.000
23	0.000000	100.000
24	0.000000	100.000
25	0.000000	100.000
26	0.000000	100.000
27	0.000000	100.000
28	0.000000	100.000
29	0.000000	100.000
30	0.000000	100.000
31	0.000000	100.000
32	0.000000	100.000
33	0.000000	100.000
34	0.000000	100.000
35	0.000000	100.000
36	0.000000	100.000

Sequence Sample Descriptions - Channel A

Row	Addend	Norm Factor
37	0.000000	100.000
38	0.000000	100.000
39	0.000000	100.000
40	0.000000	100.000
41	0.000000	100.000
42	0.000000	100.000
43	0.000000	100.000
44	0.000000	100.000
45	0.000000	100.000
46	0.000000	100.000
47	0.000000	100.000
48	0.000000	100.000
49	0.000000	100.000
50	0.000000	100.000
51	0.000000	100.000
52	0.000000	100.000
53	0.000000	100.000
54	0.000000	100.000
55	0.000000	100.000
56	0.000000	100.000
57	0.000000	100.000
58	0.000000	100.000
59	0.000000	100.000
60	0.000000	100.000
61	0.000000	100.000
62	0.000000	100.000
63	0.000000	100.000
64	0.000000	100.000
65	0.000000	100.000
66	0.000000	100.000
67	0.000000	100.000
68	0.000000	100.000
69	0.000000	100.000
70	0.000000	100.000
71	0.000000	100.000
72	0.000000	100.000
73	0.000000	100.000
74	0.000000	100.000
75	0.000000	100.000
76	0.000000	100.000
77	0.000000	100.000
78	0.000000	100.000
79	0.000000	100.000
80	0.000000	100.000
81	0.000000	100.000
82	0.000000	100.000
83	0.000000	100.000
84	0.000000	100.000
85	0.000000	100.000
86	0.000000	100.000
87	0.000000	100.000
88	0.000000	100.000
89	0.000000	100.000
90	0.000000	100.000
91	0.000000	100.000
92	0.000000	100.000
93	0.000000	100.000
94	0.000000	100.000
95	0.000000	100.000
96	0.000000	100.000
97	0.000000	100.000
98	0.000000	100.000
99	0.000000	100.000
100	0.000000	100.000
101	0.000000	100.000
102	0.000000	100.000
103	0.000000	100.000
104	0.000000	100.000
105	0.000000	100.000
106	0.000000	100.000
107	0.000000	100.000
108	0.000000	100.000
109	0.000000	100.000
110	0.000000	100.000
111	0.000000	100.000
112	0.000000	100.000
113	0.000000	100.000
114	0.000000	100.000
115	0.000000	100.000
116	0.000000	100.000
117	0.000000	100.000
118	0.000000	100.000
119	0.000000	100.000

6/25/02 8:57:45 AM Sequence: D:\TCWS Data\sequence\C-0624-2002.seq

Sequence Process Information - Channel A

Row	Vial	Inst Method	Proc Method	Calib Method
1	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
2	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
3	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
4	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
5	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
6	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
7	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
8	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
9	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
10	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
11	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
12	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
13	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
14	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
15	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
16	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
17	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
18	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
19	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
20	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
21	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
22	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
23	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
24	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
25	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
26	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
27	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
28	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
29	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
30	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
31	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
32	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
33	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
34	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
35	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
36	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
37	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
38	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
39	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
40	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
41	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
42	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
43	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
44	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
45	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
46	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
47	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
48	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
49	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
50	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
51	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
52	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
53	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
54	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
55	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
56	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
57	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
58	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
59	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
60	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
61	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
62	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
63	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
64	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
65	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
66	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
67	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
68	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
69	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
70	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
71	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
72	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
73	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
74	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
75	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
76	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
77	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
78	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
79	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
80	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
81	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
82	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
83	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
84	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
85	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426

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Sequence Process Information - Channel A

Row	Vial	Inst Method	Proc Method	Calib Method
86	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
87	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
88	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
89	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
90	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
91	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
92	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
93	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
94	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
95	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
96	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
97	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
98	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
99	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
100	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
101	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
102	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
103	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
104	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
105	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
106	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
107	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
108	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
109	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
110	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
111	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
112	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
113	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
114	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
115	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
116	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
117	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
118	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426
119	-	d:\tcws data\method\40-c5short	d:\tcws data\method\40-c5so-02-0426	d:\tcws data\method\40-c5so-02-0426

Row	Rpt Frmt File	Raw Data File	Result File	Baseline	Modified	Calib Rpt	Cal Level	Update RT	Printer	Plotter
1	d:\tcws data\method\a-c5short	CAL0624a001	CAL0624a001			-	-	-	Default	Default
2	d:\tcws data\method\a-c5short	CAL0624a002	CAL0624a002			-	-	-	Default	Default
3	d:\tcws data\method\a-c5short	CAL0624a003	CAL0624a003			-	-	-	Default	Default
4	d:\tcws data\method\a-c5short	CAL0624a004	CAL0624a004			-	-	-	Default	Default
5	d:\tcws data\method\a-c5short	CAL0624a005	CAL0624a005			-	-	-	Default	Default
6	d:\tcws data\method\a-c5short	CAL0624a006	CAL0624a006			-	-	-	Default	Default
7	d:\tcws data\method\a-c5short	CAL0624a007	CAL0624a007			-	-	-	Default	Default
8	d:\tcws data\method\a-c5short	CAL0624a008	CAL0624a008			-	-	-	Default	Default
9	d:\tcws data\method\a-c5short	CAL0624a009	CAL0624a009			-	-	-	Default	Default
10	d:\tcws data\method\a-c5short	CAL0624a010	CAL0624a010			-	-	-	Default	Default
11	d:\tcws data\method\a-c5short	CAL0624a011	CAL0624a011			-	-	-	Default	Default
12	d:\tcws data\method\a-c5short	CAL0624a012	CAL0624a012			-	-	-	Default	Default
13	d:\tcws data\method\a-c5short	CAL0624a013	CAL0624a013			-	-	-	Default	Default
14	d:\tcws data\method\a-c5short	CAL0624a014	CAL0624a014			-	-	-	Default	Default
15	d:\tcws data\method\a-c5short	CAL0624a015	CAL0624a015			-	-	-	Default	Default
16	d:\tcws data\method\a-c5short	CAL0624a016	CAL0624a016			-	-	-	Default	Default
17	d:\tcws data\method\a-c5short	CAL0624a017	CAL0624a017			-	-	-	Default	Default
18	d:\tcws data\method\a-c5short	CAL0624a018	CAL0624a018			-	-	-	Default	Default
19	d:\tcws data\method\a-c5short	CAL0624a019	CAL0624a019			-	-	-	Default	Default
20	d:\tcws data\method\a-c5short	CAL0624a020	CAL0624a020			-	-	-	Default	Default
21	d:\tcws data\method\a-c5short	CAL0624a021	CAL0624a021			-	-	-	Default	Default
22	d:\tcws data\method\a-c5short	CAL0624a022	CAL0624a022			-	-	-	Default	Default
23	d:\tcws data\method\a-c5short	CAL0624a023	CAL0624a023			-	-	-	Default	Default
24	d:\tcws data\method\a-c5short	CAL0624a024	CAL0624a024			-	-	-	Default	Default
25	d:\tcws data\method\a-c5short	CAL0624a025	CAL0624a025			-	-	-	Default	Default
26	d:\tcws data\method\a-c5short	CAL0624a026	CAL0624a026			-	-	-	Default	Default
27	d:\tcws data\method\a-c5short	CAL0624a027	CAL0624a027			-	-	-	Default	Default
28	d:\tcws data\method\a-c5short	CAL0624a028	CAL0624a028			-	-	-	Default	Default
29	d:\tcws data\method\a-c5short	CAL0624a029	CAL0624a029			-	-	-	Default	Default
30	d:\tcws data\method\a-c5short	CAL0624a030	CAL0624a030			-	-	-	Default	Default
31	d:\tcws data\method\a-c5short	CAL0624a031	CAL0624a031			-	-	-	Default	Default
32	d:\tcws data\method\a-c5short	CAL0624a032	CAL0624a032			-	-	-	Default	Default
33	d:\tcws data\method\a-c5short	CAL0624a033	CAL0624a033			-	-	-	Default	Default
34	d:\tcws data\method\a-c5short	CAL0624a034	CAL0624a034			-	-	-	Default	Default
35	d:\tcws data\method\a-c5short	CAL0624a035	CAL0624a035			-	-	-	Default	Default
36	d:\tcws data\method\a-c5short	CAL0624a036	CAL0624a036			-	-	-	Default	Default
37	d:\tcws data\method\a-c5short	CAL0624a037	CAL0624a037			-	-	-	Default	Default
38	d:\tcws data\method\a-c5short	CAL0624a038	CAL0624a038			-	-	-	Default	Default
39	d:\tcws data\method\a-c5short	CAL0624a039	CAL0624a039			-	-	-	Default	Default
40	d:\tcws data\method\a-c5short	CAL0624a040	CAL0624a040			-	-	-	Default	Default
41	d:\tcws data\method\a-c5short	CAL0624a041	CAL0624a041			-	-	-	Default	Default
42	d:\tcws data\method\a-c5short	CAL0624a042	CAL0624a042			-	-	-	Default	Default
43	d:\tcws data\method\a-c5short	CAL0624a043	CAL0624a043			-	-	-	Default	Default
44	d:\tcws data\method\a-c5short	CAL0624a044	CAL0624a044			-	-	-	Default	Default
45	d:\tcws data\method\a-c5short	CAL0624a045	CAL0624a045			-	-	-	Default	Default
46	d:\tcws data\method\a-c5short	CAL0624a046	CAL0624a046			-	-	-	Default	Default
47	d:\tcws data\method\a-c5short	CAL0624a047	CAL0624a047			-	-	-	Default	Default
48	d:\tcws data\method\a-c5short	CAL0624a048	CAL0624a048			-	-	-	Default	Default
49	d:\tcws data\method\a-c5short	CAL0624a049	CAL0624a049			-	-	-	Default	Default

6/25/02 8:57:45 AM Sequence: D:\TCWS Data\sequence\C-0624-2002.seq

Sequence Process Information - Channel A

Row	Rpt Fmt File	Raw Data File	Result File	Baseline	Modified	Calib Rpt	Cal Level	Update RT	Printer	Plotter
50	d:\tcws data\method\a-c5short	CAL0624a050	CAL0624a050			-	-	-	Default	Default
51	d:\tcws data\method\a-c5short	CAL0624a051	CAL0624a051			-	-	-	Default	Default
52	d:\tcws data\method\a-c5short	CAL0624a052	CAL0624a052			-	-	-	Default	Default
53	d:\tcws data\method\a-c5short	CAL0624a053	CAL0624a053			-	-	-	Default	Default
54	d:\tcws data\method\a-c5short	CAL0624a054	CAL0624a054			-	-	-	Default	Default
55	d:\tcws data\method\a-c5short	CAL0624a055	CAL0624a055			-	-	-	Default	Default
56	d:\tcws data\method\a-c5short	CAL0624a056	CAL0624a056			-	-	-	Default	Default
57	d:\tcws data\method\a-c5short	CAL0624a057	CAL0624a057			-	-	-	Default	Default
58	d:\tcws data\method\a-c5short	CAL0624a058	CAL0624a058			-	-	-	Default	Default
59	d:\tcws data\method\a-c5short	CAL0624a059	CAL0624a059			-	-	-	Default	Default
60	d:\tcws data\method\a-c5short	CAL0624a060	CAL0624a060			-	-	-	Default	Default
61	d:\tcws data\method\a-c5short	CAL0624a061	CAL0624a061			-	-	-	Default	Default
62	d:\tcws data\method\a-c5short	CAL0624a062	CAL0624a062			-	-	-	Default	Default
63	d:\tcws data\method\a-c5short	CAL0624a063	CAL0624a063			-	-	-	Default	Default
64	d:\tcws data\method\a-c5short	CAL0624a064	CAL0624a064			-	-	-	Default	Default
65	d:\tcws data\method\a-c5short	CAL0624a065	CAL0624a065			-	-	-	Default	Default
66	d:\tcws data\method\a-c5short	CAL0624a066	CAL0624a066			-	-	-	Default	Default
67	d:\tcws data\method\a-c5short	CAL0624a067	CAL0624a067			-	-	-	Default	Default
68	d:\tcws data\method\a-c5short	CAL0624a068	CAL0624a068			-	-	-	Default	Default
69	d:\tcws data\method\a-c5short	CAL0624a069	CAL0624a069			-	-	-	Default	Default
70	d:\tcws data\method\a-c5short	CAL0624a070	CAL0624a070			-	-	-	Default	Default
71	d:\tcws data\method\a-c5short	CAL0624a071	CAL0624a071			-	-	-	Default	Default
72	d:\tcws data\method\a-c5short	CAL0624a072	CAL0624a072			-	-	-	Default	Default
73	d:\tcws data\method\a-c5short	CAL0624a073	CAL0624a073			-	-	-	Default	Default
74	d:\tcws data\method\a-c5short	CAL0624a074	CAL0624a074			-	-	-	Default	Default
75	d:\tcws data\method\a-c5short	CAL0624a075	CAL0624a075			-	-	-	Default	Default
76	d:\tcws data\method\a-c5short	CAL0624a076	CAL0624a076			-	-	-	Default	Default
77	d:\tcws data\method\a-c5short	CAL0624a077	CAL0624a077			-	-	-	Default	Default
78	d:\tcws data\method\a-c5short	CAL0624a078	CAL0624a078			-	-	-	Default	Default
79	d:\tcws data\method\a-c5short	CAL0624a079	CAL0624a079			-	-	-	Default	Default
80	d:\tcws data\method\a-c5short	CAL0624a080	CAL0624a080			-	-	-	Default	Default
81	d:\tcws data\method\a-c5short	CAL0624a081	CAL0624a081			-	-	-	Default	Default
82	d:\tcws data\method\a-c5short	CAL0624a082	CAL0624a082			-	-	-	Default	Default
83	d:\tcws data\method\a-c5short	CAL0624a083	CAL0624a083			-	-	-	Default	Default
84	d:\tcws data\method\a-c5short	CAL0624a084	CAL0624a084			-	-	-	Default	Default
85	d:\tcws data\method\a-c5short	CAL0624a085	CAL0624a085			-	-	-	Default	Default
86	d:\tcws data\method\a-c5short	CAL0624a086	CAL0624a086			-	-	-	Default	Default
87	d:\tcws data\method\a-c5short	CAL0624a087	CAL0624a087			-	-	-	Default	Default
88	d:\tcws data\method\a-c5short	CAL0624a088	CAL0624a088			-	-	-	Default	Default
89	d:\tcws data\method\a-c5short	CAL0624a089	CAL0624a089			-	-	-	Default	Default
90	d:\tcws data\method\a-c5short	CAL0624a090	CAL0624a090			-	-	-	Default	Default
91	d:\tcws data\method\a-c5short	CAL0624a091	CAL0624a091			-	-	-	Default	Default
92	d:\tcws data\method\a-c5short	CAL0624a092	CAL0624a092			-	-	-	Default	Default
93	d:\tcws data\method\a-c5short	CAL0624a093	CAL0624a093			-	-	-	Default	Default
94	d:\tcws data\method\a-c5short	CAL0624a094	CAL0624a094			-	-	-	Default	Default
95	d:\tcws data\method\a-c5short	CAL0624a095	CAL0624a095			-	-	-	Default	Default
96	d:\tcws data\method\a-c5short	CAL0624a096	CAL0624a096			-	-	-	Default	Default
97	d:\tcws data\method\a-c5short	CAL0624a097	CAL0624a097			-	-	-	Default	Default
98	d:\tcws data\method\a-c5short	CAL0624a098	CAL0624a098			-	-	-	Default	Default
99	d:\tcws data\method\a-c5short	CAL0624a099	CAL0624a099			-	-	-	Default	Default
100	d:\tcws data\method\a-c5short	CAL0624a100	CAL0624a100			-	-	-	Default	Default
101	d:\tcws data\method\a-c5short	CAL0624a101	CAL0624a101			-	-	-	Default	Default
102	d:\tcws data\method\a-c5short	CAL0624a102	CAL0624a102			-	-	-	Default	Default
103	d:\tcws data\method\a-c5short	CAL0624a103	CAL0624a103			-	-	-	Default	Default
104	d:\tcws data\method\a-c5short	CAL0624a104	CAL0624a104			-	-	-	Default	Default
105	d:\tcws data\method\a-c5short	CAL0624a105	CAL0624a105			-	-	-	Default	Default
106	d:\tcws data\method\a-c5short	CAL0624a106	CAL0624a106			-	-	-	Default	Default
107	d:\tcws data\method\a-c5short	CAL0624a107	CAL0624a107			-	-	-	Default	Default
108	d:\tcws data\method\a-c5short	CAL0624a108	CAL0624a108			-	-	-	Default	Default
109	d:\tcws data\method\a-c5short	CAL0624a109	CAL0624a109			-	-	-	Default	Default
110	d:\tcws data\method\a-c5short	CAL0624a110	CAL0624a110			-	-	-	Default	Default
111	d:\tcws data\method\a-c5short	CAL0624a111	CAL0624a111			-	-	-	Default	Default
112	d:\tcws data\method\a-c5short	CAL0624a112	CAL0624a112			-	-	-	Default	Default
113	d:\tcws data\method\a-c5short	CAL0624a113	CAL0624a113			-	-	-	Default	Default
114	d:\tcws data\method\a-c5short	CAL0624a114	CAL0624a114			-	-	-	Default	Default
115	d:\tcws data\method\a-c5short	CAL0624a115	CAL0624a115			-	-	-	Default	Default
116	d:\tcws data\method\a-c5short	CAL0624a116	CAL0624a116			-	-	-	Default	Default
117	d:\tcws data\method\a-c5short	CAL0624a117	CAL0624a117			-	-	-	Default	Default
118	d:\tcws data\method\a-c5short	CAL0624a118	CAL0624a118			-	-	-	Default	Default
119	d:\tcws data\method\a-c5short	CAL0624a119	CAL0624a119			-	-	-	Default	Default

Table 1: ENVIRONMENTAL REPORTING LIMITS (RL) FOR DEQ-DWRP LABORATORY SECTION

Pesticides Method 608/8081	Water Reporting Limits (ug/L)	Sediment/Soil Reporting Limits (ug/Kg)	Oil Reporting Limits (mg/Kg)	Water Containers & Hold Times	Sediment/Soil Containers & Hold Times	Oil Containers
Aldrin	0.02	20	0.2	2 - 1000mL glass	1 - 8 ounce glass	10mL in glass
a-BHC	0.02	20	0.2	amber bottles	14 days HT	"
b-BHC	0.02	20	0.2	7 days HT	"	"
d-BHC	0.02	20	0.2	"	"	"
g-BHC (lindane)	0.02	20	0.2	"	"	"
BP-6 (PPB)	0.05	250	0.7	"	"	"
a-Chlordane	0.02	50	0.2	"	"	"
g-Chlordane	0.02	50	0.2	"	"	"
4,4'-DDD	0.02	20	0.2	"	"	"
4,4'-DDE	0.02	20	0.2	"	"	"
4,4'-DDT	0.02	20	0.2	"	"	"
Dieldrin	0.02	20	0.2	"	"	"
Endosulfan I	0.02	20	0.2	"	"	"
Endosulfan II	0.05	20	0.4	"	"	"
Endosulfan Sulfate	0.05	20	0.4	"	"	"
Endrin	0.02	20	0.2	"	"	"
Endrin Aldehyde	0.05	20	0.4	"	"	"
Endrin Ketone	0.05	20	0.4	"	"	"
Heptachlor	0.02	20	0.4	"	"	"
Heptachlor epoxide	0.02	20	0.4	"	"	"
Hexabromobenzene	0.02	100	0.2	"	"	"
Methoxychlor	0.05	50	0.4	"	"	"
Mirex	0.02	50	0.2	"	"	"
Toxaphene	0.1	170	10	"	"	"

PCBs Method 608/8082	Water Reporting Limits (ug/L)	Sediment/Soil Reporting Limits (ug/Kg)	Oil Reporting Limits (mg/Kg)	Water Containers & Hold Times	Sediment/Soil Containers & Hold Times	Oil Containers
PCB-1016	0.1	100	1.0	2 - 1000mL glass	1 - 8 ounce glass	10mL in glass
PCB-1221	0.1	100	1.0	amber bottles	14 days HT	"
PCB-1232	0.1	100	1.0	7 days HT	"	"
PCB-1242	0.1	100	1.0	"	"	"
PCB-1248	0.1	100	1.0	"	"	"
PCB-1254	0.1	100	1.0	"	"	"
PCB-1260	0.1	100	1.0	"	"	"
PCB-1262	0.1	100	1.0	"	"	"
PCB-1268	0.1	100	1.0	"	"	"

Table 1: ENVIRONMENTAL REPORTING LIMITS (RL) FOR DEQ-DWRP LABORATORY SECTION

NPDES Scan 3 Method 608	Water Reporting Limits (ug/L)	Water Containers & Hold Times
Aldrin	0.01	2 - 1000mL glass
a-BHC	0.01	amber bottles
b-BHC	0.01	7 days HT
d-BHC	0.01	"
g-BHC (lindane)	0.01	"
BP-6 (PBB)	0.05	"
a-Chlordane	0.01	"
g-Chlordane	0.01	"
4,4'-DDD	0.05	"
4,4'-DDE	0.01	"
4,4'-DDT	0.01	"
Dieldrin	0.01	"
Endosulfan I	0.01	"
Endrin	0.01	"
Heptachlor	0.01	"
Heptachlor epoxide	0.01	"
Hexabromobenzene	0.01	"
Hexachlorobenzene	0.01	"
Hexachlorobutadiene	0.01	"
Hexachlorocyclopentadiene	0.01	"
Methoxychlor	0.05	"
Mirex	0.01	"
PCB 1016	0.1	"
PCB 1221	0.1	"
PCB 1232	0.1	"
PCB 1242	0.1	"
PCB 1248	0.1	"
PCB 1254	0.1	"
PCB 1260	0.1	"
PCB 1262	0.1	"
PCB 1268	0.1	"
Pentachlorobenzene	0.01	"
Pentachloronitrobenzene	0.01	"
Toxaphene	0.1	"
1,2,3,4-Tetrachlorobenzene	0.01	"
1,2,4,5-Tetrachlorobenzene	0.01	"

This scan is available for NPDES samples only.

Gas Chromatograph Operating Conditions for Pest/PCB
(Adjusted As Needed)

6890 GC #07

Inlet
Mode-Splitless
Purge time -0.9 mm
Purge flow- 50ml/min
Total flow- 60.5ml/min
Pulsed pressure- 16.3
Pulse time- 1.0 min
Temp- 190 deg

Column
30 meter, .25mm, .25 um
Flow- 4.5ml/min
Velocity- varies
Mode- constant flow

Detectors- microECD
Make-up gas- N2
Flow- 25 ml/min
Temp- 325 deg.

Temp Program
Start Temp- 60 deg for 1 min.
Init. rate- 20 deg/min 160 deg for 0 min.
Rate 2- 5 deg/min to 24O deg for 0 min.
Rate 3- 3 deg/min to 255 deg for 0 min.
Rate 4- 5 deg/min to 275 deg for 0 min.
Rate 5- 25 deg/min to 295 deg for 4 min
Run time - 29 min.

Pressure at 60 deg- 7.3
Range- 7
Attn- 0

6890 GC #40

Inlet
Mode-Splitless
Purge time -0.9 min
Purge flow- 50m1/min
Total flow- 61.7ml/min
Pulsed pressure- 16.3
Pulse time- 1.0 min
Temp- 190 deg

Column
30 meter, .25mm, .25 um
Flow- 5.0m1/min
Velocity- varies
Mode- constant flow

Detectors- microECD
Make-up gas- N2
Flow- 60 ml/mm
Temp- 325 deg.

Temp Program
Start Temp-50 deg for 0.5 min.
Init. rate-25 deg/min to 200deg for 0.5 min.
Rate 2- 2 deg/min to 220 deg for 3 min.
Rate 3- 6 deg/min to 270 deg for 0.5 min
Rate 4- 25 deg/min to 300 for 10 min.
Run time - 36.83 min.

Range- 7
Attn- 0

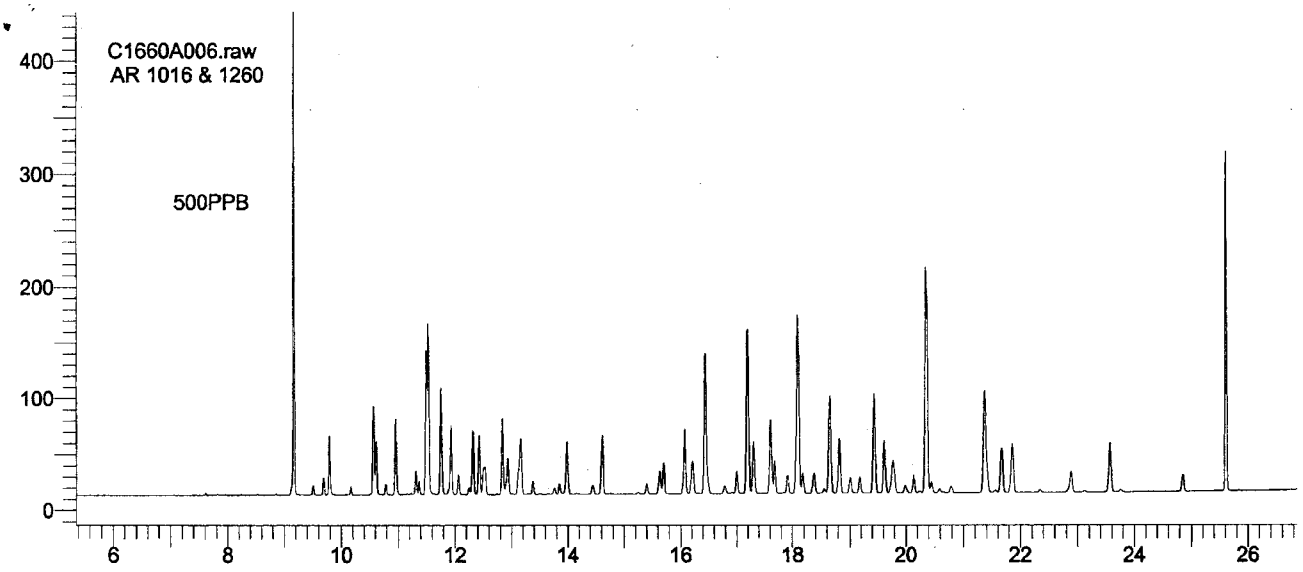
5890 GC #04

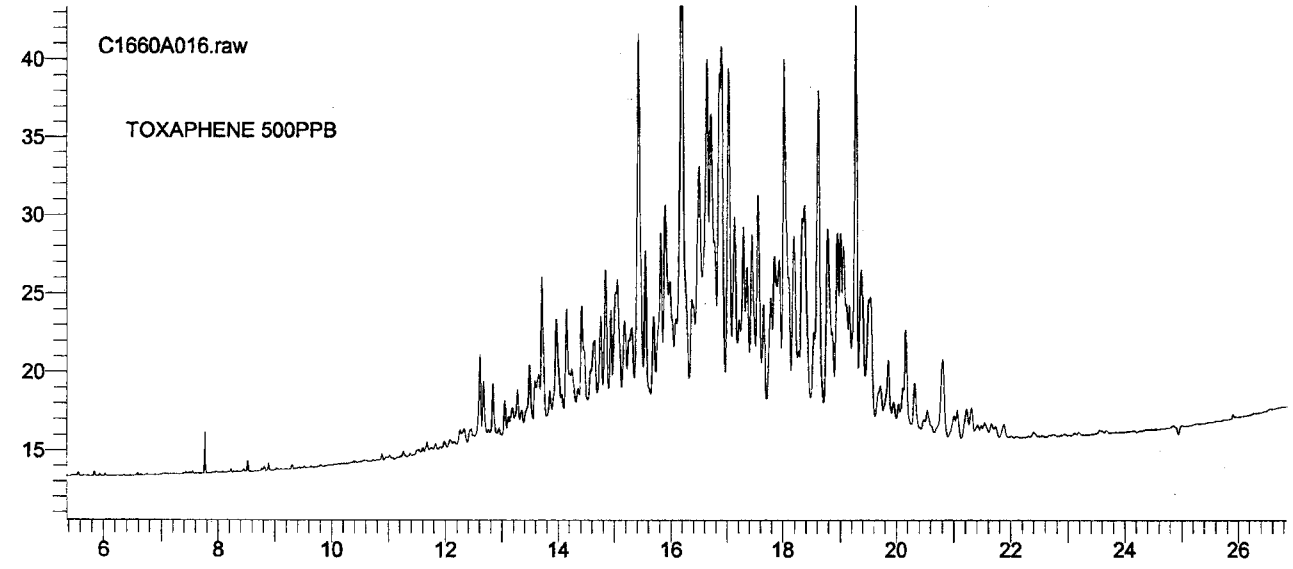
Inlet
Mode-Splitless
Purge time - 1.0 min
Head Pressure - l3psig
Temp- 190 deg

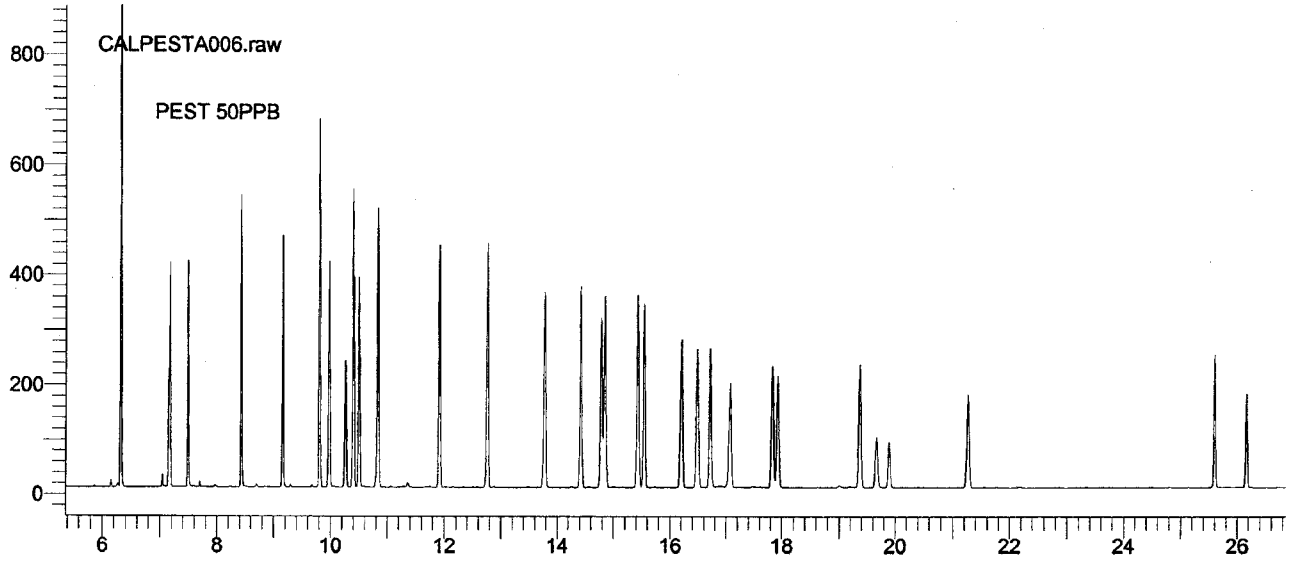
Column
30 meter, .25mm, .25 um
Flow- variable
Mode- constant pressure

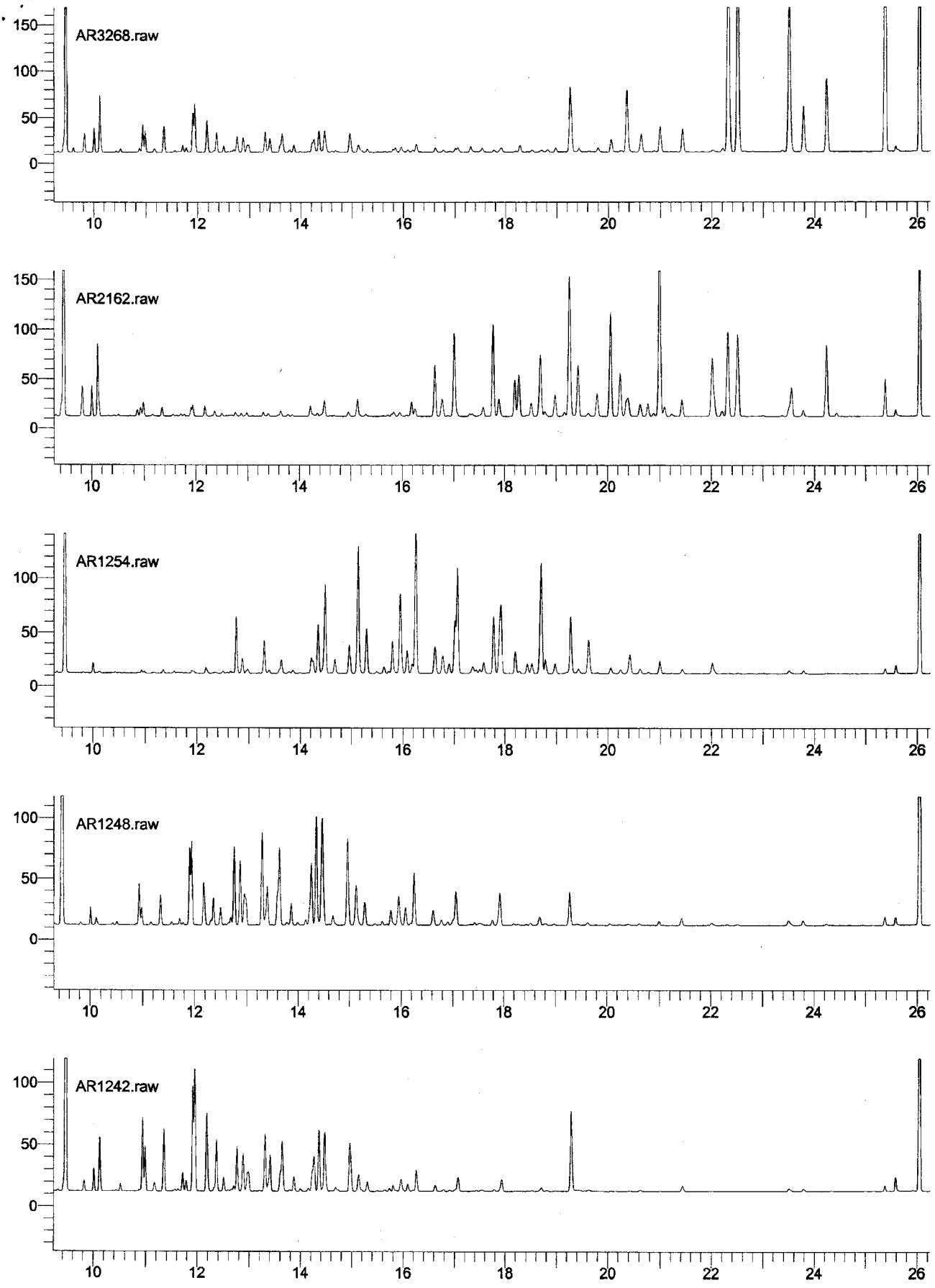
Detectors-ECD
Make-up gas- N2
Flow- 50 ml/mm
Temp- 350 deg.

Temp Program
Start Temp- 55 deg for 0.5 min.
Init: rate- 25 deg/min to 140 deg for 0.5 min.
Rate 2- 3 deg/min to 250deg for 0.5 min.
Rate 3- 25 deg/min to 300 deg for 4.6 min.
Run time - 48 min.
Range- col A is 0, col B is 2
Attn- 0


C1660A006.raw
AR 1016 & 1260
500PPB
400
300
200
100
0
6
8
10
12
14
16
18
20
22
24
26

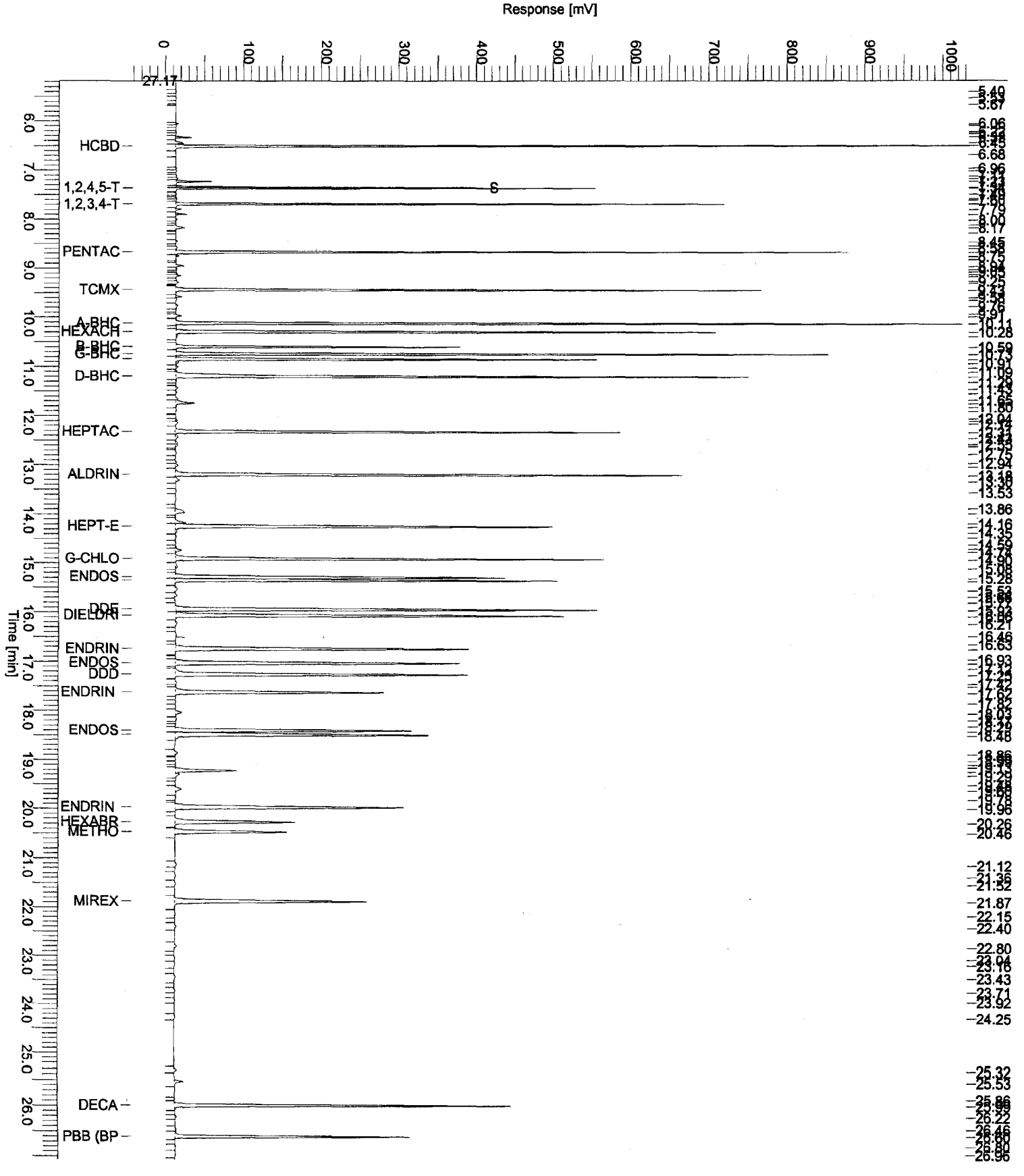
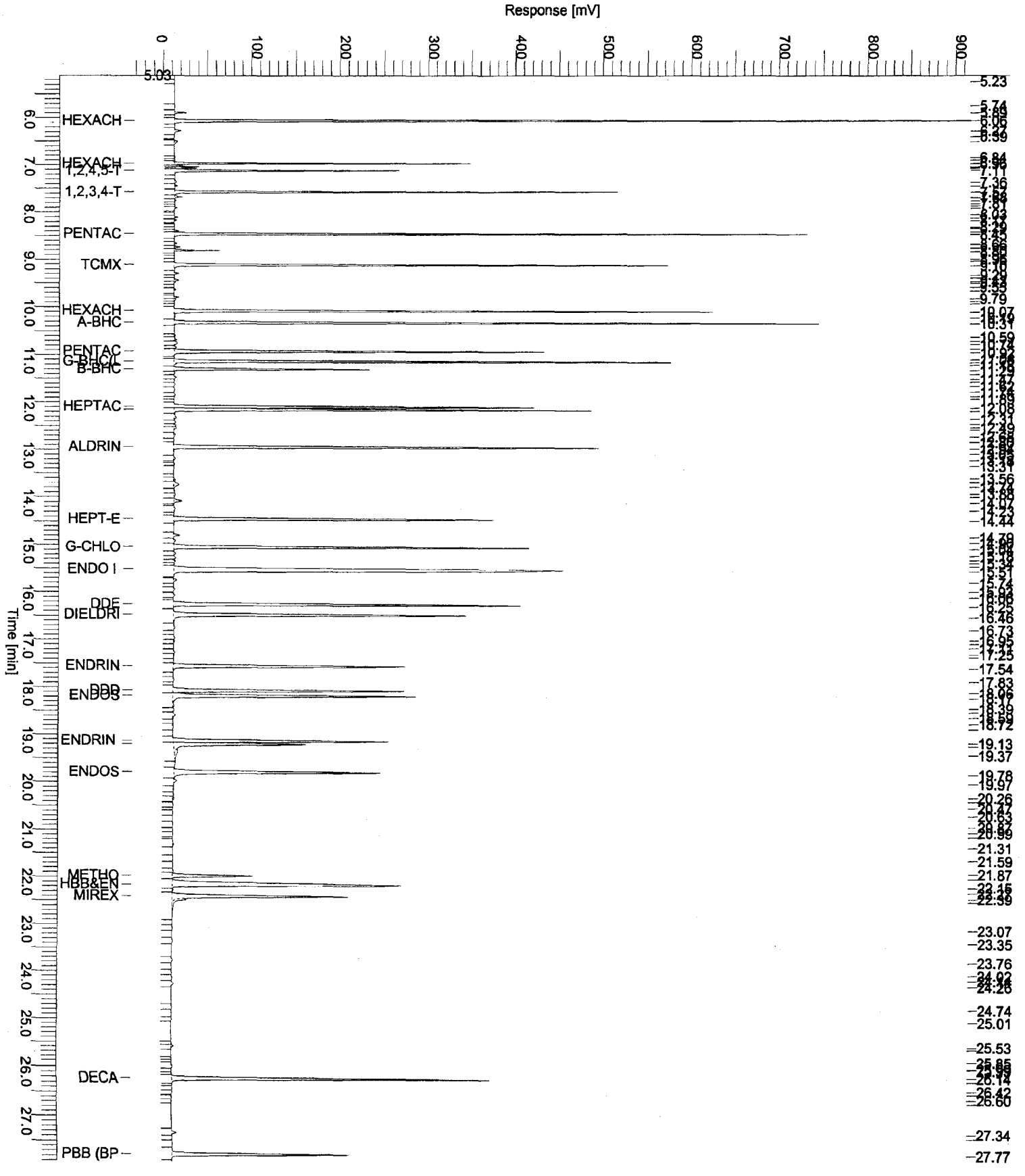

C1660A016.raw
TOXAPHENE 500PPB
40
35
30
25
20
15
6
8
10
12
14
16
18
20
22
24
26


CALPESTA006.raw
PEST 50PPB
800
600
400
200
0
6
8
10
12
14
16
18
20
22
24
26


AR3268.raw
AR2162.raw
AR1254.raw
AR1248.raw
AR1242.raw
0
50
100
150
10
12
14
16
18
20
22
24
26

Chromatogram-Ch. A

Sample Name : PEST SE-744 125PPB Sample #: 03 Page 1 of 1
FileName : D:\TCWS Data\data\5001a003.raw
Date : 10/8/02 4:43:52 PM
Method :
Time of Injection: 10/1/02 2:02:33 PM
Start Time : 5.18 min End Time : 27.07 min Low Point : -41.94 mV High Point : 1033.25 mV
Plot Offset: -41.94 mV Plot Scale: 1075.2 mV



Sample Name : PEST SE-744 125PPB Sample #: 03 Page 1 of 1
FileName : D:\TCWS Data\data\5001b003.raw
Date : 10/8/02 4:44:45 PM
Method : 7-pestb-020925.mth Time of Injection: 10/1/02 2:02:33 PM
Start Time : 5.11 min End Time : 27.93 min Low Point : -33.22 mV High Point : 916.41 mV
Plot Offset: -33.22 mV Plot Scale: 949.6 mV