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SOLUTIA - 199

"Smith, Steven D"
<sdsmit@solutia.com>
06/29/2005 08:58 AM

To Kenneth Bardo/R5/USEPA/US@EPA
cc "Yare, Bruce S" <bsyare@solutia.com>
bcc

Subject W G Krummrich - CMS

History: This message has been forwarded.

Ken;

We were discouraged to receive your email last week transmitting your letter dated June 23, 2005.

As you know, for the last several months we have been discussing and refining the appropriate work plans for an In-Situ Thermal Desorption (ISTD) and an Enhanced Aerobic Bioremediation (EAB) bench scale test for WGK soils. Your email of June 17th, 2005 indicated that, while EPA was not formally approving the work plans, Solutia should begin to carry out this work immediately. Based on this June 17th, 2005 email, Solutia directed our consultants to begin work immediately in lining up drillers and secondary consultants such as Kemron Environmental Services.

However, your letter of June 23, 2005 sets forth significant new requirements and issues related to the above tests (over and beyond the June 17th, 2005 email). Some of the more significant, newly identified issues raised are as follows:

ISTD Bench Scale Study

- The 6/23/05 comments state that the ISTD bench study must also "evaluate the potential performance of applicable technologies and generate data for scale up to a field pilot test of the most favorable technology or technologies". The study we have proposed and discussed over the last several months does not meet this requirement. Therefore, it makes sense that we revisit the very intent of the ISTD bench study before we commence such work.

- The 6/23/2005 letter adds a new requirement for two additional tests on saturated soil samples from the SHU ; one at 132 degrees C and another at 200 degrees C. We believe these tests duplicate the testing being done on the unsaturated soil samples at these temperatures. Furthermore, any water in the unsaturated test will be long gone after 72 hours at 100 degrees C.

- The 6/23/2005 letter asks for a number of additional documents to be developed and submitted, including:

  - Data Quality Objectives
  - New Lab SOPs
  - Rationale for the selection of target temperatures
  - Provide air flow rates through the test cylinder containing the soil sample during heating
  - Modify Field Sampling Plan, clarifying various QA/QC issues

EAB Bench Scale Study

- New Lab SOPs, Data Quality Objective, and various modifications and clarifications to the work plan.

Our suggestion is that we go ahead and initiate the EAB Bench Scale Study in hope of being able to provide the additional information requested to EPA's satisfaction during the bench scale time period. This will result in additional mobilization costs for Solutia, but will initiate this critical path issue.

Obviously we need to reconcile some significant new issues before undertaking the ISTD testing. Since the EAB tests takes longer to perform than the ISTD tests , we hopefully can resolve these issues and not delay the overall schedule.

We are still targeting the Plume Stability Monitoring Plan for submittal on July 5th, 2005.

I suggest that we schedule a meeting during the week of July 11th or 18th. At that meeting we can hopefully resolve the ISTD issues and also discuss any issues related to the soil sampling plan or the Plume Stability Monitoring Plan.

If this seems reasonable, please advise. When Bruce Yare gets back in town I will find a couple of potential dates where we could fly up to Chicago to meet.

Steve

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July 5, 2005

Mr. Kenneth Bardo
U.S. EPA Region V
Corrective Action Section
Enforcement Compliance Branch
77 West Jackson Boulevard DE-J9
Chicago, IL 60604-3507

Re: Solutia Inc. – W. G. Krummrich Plant, RCRA
Supplemental Soil and Groundwater Sampling Plan

Dear Ken:

Enclosed is the Supplemental Soil and Groundwater Sampling Plan for the W. G. Krummrich Plant.

We plan to go ahead and initiate the sampling specified in this plan later this month.
Any questions, please call.

Sincerely,

Steven D. Smith
Project Manager

cc: Distribution List on Following Page

Distribution list for the July 5th, 2005 Supplemental Soil and Groundwater Sampling Plan

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TECHNCIAL TEAM		
Bob Billman	URS, 1010 Highlands Plaza Drive West, Suite 300, St. Louis, MO 63110	1 Copy
Chuck Newell	Groundwater Services Inc., 2211 Norfolk, Suite 1000, Houston, TX 77098	1 Copy
Gale Hoffnagle	TRC, 5 Waterside Crossing, Windsor, CT 06095-1563	1 Copy
MONSANTO		
Glen Kurowski	Monsanto, 800 N. Lindbergh Blvd, St. Louis, MO 63141	1 Copy
Linda Tape	Husch, Eppenberger, 190 Carondelet Plaza, Suite 600, St. Louis, MO 63105-3441	1 Copy

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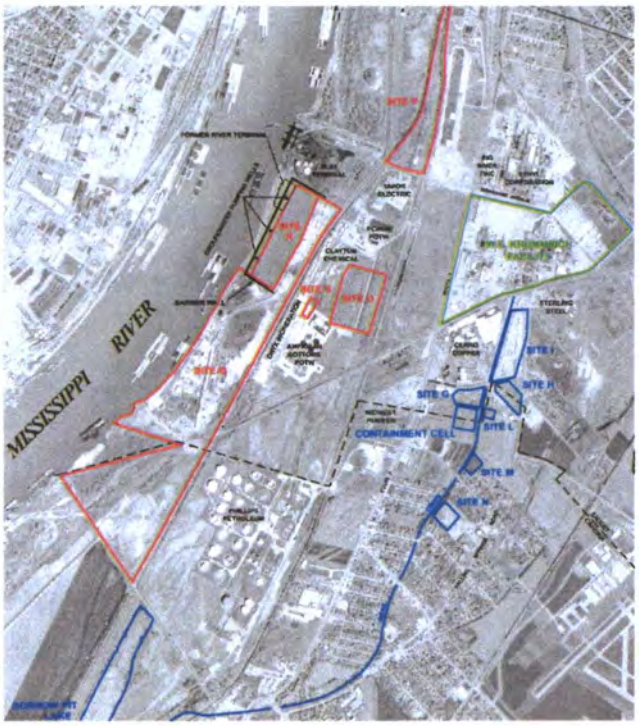
SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING PLAN

SOLUTIA INC.
W.G. KRUMMRICH FACILITY
SAUGET, ILLINOIS



Prepared by:
Solutia Inc.
575 Maryville Centre Dr.
St. Louis, Missouri 63141

URS

July 2005

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Appendix B Laboratory Standard Operating Procedures (SOPs)

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1.0 INTRODUCTION

On May 3, 2000, USEPA executed a Resource Conservation and Recovery Act (RCRA) 3008(h) Administrative Order on Consent for Solutia Inc.'s W.G. Krummrich facility in Sauget, Illinois (**Figures 1.1 and 1.2**). Solutia Inc. signed the Administrative Order on Consent, Docket No. R8H-5-00-003, on May 26, 2000. Sections VI.1a, 1b, 2, 3 and 5, respectively, required Solutia to submit a Description of Current Conditions Report, investigate the nature and extent of any releases at or from the W.G. Krummrich facility, stabilize groundwater migration and show that any discharge of groundwater to surface water is either insignificant or currently acceptable, control completed pathway human exposures to contamination and propose final corrective measures for the site.

To fulfill the requirements of the AOC Solutia submitted a Description of Current Conditions Report, performed site investigations for air, soil, DNAPL and groundwater, completed Environmental Indicator Determinations for Migration of Contaminated Groundwater under Control (CA750) and Current Human Exposure Under Control (CA725) and submitted a Final Corrective Measures Study as summarized in the following table:

Summary of Work Performed to Fulfill the Requirements of the W.G. Krummrich RCRA AOC (Docket No. R8H-5-00-003)

• Description of Current Conditions Report	August 1, 2000
• Sediment, Surface Water and Fish Tissue Sampling	October and November 2000
• Ecological Risk Assessment	June 1, 2001
• CA750 Migration of Contaminated Groundwater Under Control Environmental Indicator Determination	May 26, 2004
• CA725 Current Human Exposure Under Control Environmental Indicator Determination	May 26, 2004
• Air, Soil, DNAPL and Groundwater Investigation	2003 and 2004
• Corrective Measures Study	August 27, 2004

In addition to these actions, Solutia implemented or planned a number of removal and remedial actions at Sauget Area 1, Sauget Area 2 and the W.G. Krummrich Facility prior to and after the May 26, 2000 RCRA AOC. A time line of the various removal actions and remedial actions and estimated expenditures for each action are given below:

Time Line of Sauget Area 1, Sauget Area 2 and W.G. Krummrich Removal/Remedial Actions and Estimated Expenditures

Sauget Area 1	2001	Dead Creek Culvert Replacement Removal Action	$750,000
	2002	Dead Creek Time Critical Sediment Removal Action	12,300,000
	2004	Dead Creek Segment B, D and F Soil Removal Action Plan	
Sauget Area 2	1979	Site R Capping	
	1985	Site R Riverbank Stabilization	750,000
	2003/4	Groundwater Migration Control System	25,400,000
W.G. Krummrich	1987	Route 3 Drum Site Impermeable Cap	
	2000	Sewer System Improvements	17,100,000
	2001	Chlorobenzene Process Area Spill	
	2003	Plant Process Area Permeable Covers	310,000
		Estimated Total Expenditure	$56,610,000

In comments provided by USEPA in May 2005 regarding the Corrective Measures Study, the Agency identified specific supplemental investigations necessary to further characterize potential source areas and associated risks. These areas include the Route 3 Drum Site, Lot F, Former Chlor-Alkali Production Area, North Central Plant Process Area, and the Former Coal Storage Area. Solutia is providing this

supplemental soil and groundwater sampling work plan, which describes the planned sampling at these select locations as directed by the Agency.

This Supplemental Soil and Groundwater Sampling Work Plan includes the following sections:

Section 1.0	**Introduction**
Section 2.0	**Supplemental Groundwater Investigation**
Section 3.0	**Supplemental Soil Investigation**
Section 4.0	**Data Management, Evaluation and Reporting**
Section 5.0	**Schedule**
Section 6.0	**References**

The sections describe the locations for sampling, sampling procedures, laboratory analytical program, data validation, and reporting. Figures and Tables follow the text. Field Standard Operating Procedures (SOPs) and laboratory SOPs are includes in Appendices A and B, respectively.

2.0 SUPPLEMENTAL GROUNDWATER INVESTIGATION

2.1 Objectives and Approach of the Supplemental Groundwater Investigation

The Route 3 Drum Site [Solid Waste Management Unit (SWMU) 27], located in the southwest corner of Lot F, was used as a drum disposal site before 1946. The Route 3 Drum Site was the subject of several interim actions from January 1985 to October 1987 consisting of excavation and off-site disposal, capping and fencing.

As directed by USEPA, Solutia plans additional characterization of groundwater quality at the Route 3 Drum Site to determine if the work performed to date, in addition to the groundwater collection provided by the Sauget Area 2 Groundwater Migration Control System, is sufficient to protect human health and the environment. The additional characterization includes redeveloping and sampling eight existing monitoring wells. The wells are shown on **Figure 2.1**.

2.2 Monitoring Well Redevelopment

Monitoring wells GM-8, GM-31A, GM-31B, GM-31C, GM-54A, GM-54B, GM-58A, and GM-59A will be redeveloped to remove any accumulated sediment using a conventional groundwater pump, air-lift system, or equivalent methods suitable for well development. Construction information for the monitoring wells is shown on **Table 2.1**. Before well redevelopment begins, the wells will be inspected for security, damage, and evidence of tampering. If damage or tampering is evident, the project manager will be contacted for guidance. Groundwater depth and total well depth (and the presence of any non-aqueous phase liquids (NAPL)) will be measured to the nearest 1/100 ft using an electronic interface probe and documented. Each monitoring well will be redeveloped until a minimum of five well volumes have been removed and pH, specific conductance, and temperature readings stabilize within 10% over a minimum of two successive readings. In addition, the turbidity of the development water will be observed to see if suspended fines have been removed. The field parameters will be recorded on monitoring well sampling sheets during redevelopment. **SOP-1** described the well redevelopment procedures.

2.3 Groundwater Sampling Procedures

Groundwater level measurements and samples will be obtained from the eight monitoring wells listed above and illustrated on **Figure 2.1**. Groundwater level measurements will be obtained in accordance with **SOP-2**. Groundwater samples will be analyzed for SVOCs (Method 8270C) and PCBs (Method 8082).

Groundwater samples will be collected using low-flow methodologies including a flow-through cell. General procedures for low-flow sample collection are described below. Additional details are included in **SOP-3**. The groundwater sampling will proceed from the least impacted wells to the most impacted, based on available information. Equipment used for sampling that could contact groundwater will be properly decontaminated before each use. Field instruments will be calibrated prior to use in accordance with the manufacturer's specifications.

Clean plastic sheeting will be placed around the well and ambient volatile organic compound (VOC) background levels in the immediate vicinity of the well will be measured (i.e., using a photoionization detector (PID) or a flame ionization detector (FID)). Once the well cap is removed, VOCs will be

measured at the rim of the well and the readings recorded in the logbook or on the groundwater sampling form.

Immediately prior to sampling, groundwater depths (and the presence of any NAPL) will be measured to the nearest 1/100 ft using an electronic interface probe and documented. If NAPL is present, efforts will be made to collect water above or below the NAPL. The depth to the bottom of each well will be measured immediately after sampling, to minimize disturbing the water column.

The monitoring wells will be purged using a conventional groundwater pump, suitable for low flow applications (i.e., bladder pump [or equivalent]). Prior to purging, the pump will slowly be lowered to a depth in the well as described in the SOP. When purging first begins, the pump flow rate will be started at approximately 100mL/min or the lowest flow rate possible. Water level measurements and flow rate measurements will be taken every 2 minutes until they indicate that significant drawdown within the well is not occurring. Measurements will be scaled back to every five minutes when drawdown reaches equilibrium. Ideally, drawdown will be limited to 25% of the distance between the top of the screen and the pump intake. If significant drawdown occurs, the well will be pumped dry. After being pumped dry, the well will be periodically gauged until the water level has recharged to approximately 90% of the original, static level prior to sampling. If in 24 hours the well has not reached 90% static recovery, the well will be sampled.

Each monitoring well will be purged until pH, specific conductance, dissolved oxygen and oxygen reduction potential stabilize over a minimum of three successive flow-through cell volumes. In addition, temperature and turbidity will be measured but not used as sampling criteria. The field parameters will be measured and recorded on monitoring well sampling sheets during purging. The allowable ranges for the criteria used to determine stabilization is provided below:

- pH +/- 0.2 units
- Conductivity +/- 3%
- DO +/- 10% or +/-0.2 mg/L, whichever is greatest
- ORP +/- 20 mV

After the relevant parameters have stabilized, the flow-through cell will be bypassed for sampling. Groundwater samples will be collected at a flow rate not greater than 0.5 L/min (to minimize aeration) using the same pump used for purging. Personnel conducting the groundwater sampling will wear clean disposable protective gloves. The appropriate sample containers as described in **Table 2.2** will be filled in the order below:

- Semivolatile organic compounds
- Polychlorinated biphenyls

To verify field and laboratory procedures, quality assurance/quality control (QA/QC) samples consisting of duplicate samples, matrix spike/matrix spike duplicate (MS/MSD) samples and equipment blanks will be collected and submitted to the laboratory. QA/QC samples will be collected at a frequency of 10% for duplicates and blanks and 5% for MS/MSDs.

Equipment test checks are to be used to ensure QA/QC for the equipment used in field work. In order to reduce the potential for exposure to hazardous materials and limit the possibility of cross contamination of samples, field personnel and equipment will comply with decontamination and IDW procedures as described in the following section.

For proper identification in the field and proper tracking by the analytical laboratory, investigative and QC samples will be labeled in a clear and consistent fashion. Sample labels will be waterproof, or sample containers will be sealed in plastic bags.

A completed sample label will be attached to each investigative or QC sample. The following will be recorded with permanent ink on sample labels by the field sampling team:

- Project name and number,
- Sample number identification,
- Initials of sampler,
- Sampling location,
- Required analysis,
- Date and time of sample collection,
- Space for laboratory sample number, and
- Preservative used, if applicable.

The sample identification system for groundwater sampling will involve the following nomenclature "AAA-BB" where:

"AAA" will denote the monitoring well

- GM8- Monitoring Well Location

"BB" will denote QA/QC sample

- EB- equipment blank
- AD- analytical duplicate
- MS or MD – Matrix Spike or Matrix Duplicate
- TB- Trip Blank

All samples will be placed on ice inside a cooler immediately following sampling. Sampling containers will be packed in such a way as to help prevent breakage and cross-contamination. Samples will be shipped in coolers, each containing a chain-of-custody form and ice packs to maintain inside temperature at approximately 4°C. Sample coolers will then be sealed between the lid and sides of the cooler with a custody seal prior to shipment. The custody seal will be an adhesive-backed tape that easily rips if it is disturbed. Samples will be shipped to the laboratory by common overnight carrier.

Sample transportation will comply with U.S. Department of Transportation and IICAO/IATA (1999)

regulations. Special sampling packing provisions will be made for samples requiring additional protection.

Field personnel will maintain a sampling log sheet containing information sufficient to allow reconstruction of the sample collection and handling procedures at a later time. Chain-of-custody procedures will be instituted and followed throughout the sampling activities. Samples are physical evidence and will be handled according to strict chain-of-custody protocols. The field sampler is personally responsible for the care and custody of the sample until transferred.

Field personnel will record the following information with permanent ink on the chain-of-custody:

- Project identification and number,
- Sample description/location,
- Required analysis,
- Date and time of sample collection,
- Type and matrix of sample,
- Number of sample containers,
- Analysis requested/comments,
- Sampler signature/date/time, and
- Air bill number.

A chain-of-custody document providing all information, signatures, dates, and other information, as required on the example chain-of-custody form included in **SOP-4** will be completed by the field sampler and provided for each sample cooler.

Samples will remain in the custody of the sampler until transfer of custody is completed. Transfer consists of:

- Delivery of samples to the laboratory sample custodian, and
- Signature of the laboratory sample custodian on the chain-of-custody document as receiving the samples, and signature of sampler, as relinquishing the samples.

If a carrier is used to take samples between the sampler and the laboratory, a copy of the air bill must be attached to the chain-of-custody to maintain proof of custody.

When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the chain-of-custody. The field sampler will sign the chain-of-custody form when relinquishing custody, make a copy to keep with the field logbook, and include the original form in an air-tight plastic bag in the sample cooler with the associated samples.

The laboratory will assign a number for each sample upon receipt. That sample number will be placed on the sample label.

Additional information concerning sample handling, preservation and tracking is contained in **SOPs** 4 and

5.

2.4 Field Documentation

URS personnel will keep a bound field notebook while performing sampling and oversight activities on-site. Forms that will be used include: chain-of-custody, development sheets, sampling data sheets, and field logbooks. The field logbooks will record the following:

- Changes to the plan;
- Personnel conducting the site activities, their arrival and departure times and their destination at the site;
- Location where the work was performed;
- Date, time, weather conditions, equipment, and personnel on site;
- Daily information such as:
  - Work accomplished and the current site status,
  - Equipment calibrations, repairs and results, and
  - Site work zones;
- Specific work activities conducted such as
  - Work zone and headspace readings; and
- Incidents and unusual activities that occur on the site such as, but not limited to, accidents, breaches of security, injuries, equipment failures, or weather related problems.

The following sampling-related information will be recorded in the field logbook by the field sampling team:

- Sample number,
- Project identification,
- Sampling location,
- Required analysis,
- Date and time of sample collection,
- Type and matrix of sample,
- Sampling technique,
- Preservative used, if applicable,
- Sampling conditions,
- Observations,
- Initials of the sampler,

- Samples collected,
- Water level,
- Product level if necessary, and
- Depth to bottom of well.

Entries will be signed and dated, and any entry which is to be deleted will have a single cross out which is signed and dated.

Procedures to evaluate field data for this project primarily include checking for transcription errors on the part of field crew members and review of field notebooks. This task will be the responsibility of the URS Field Leader, who will otherwise not participate in making any of the field measurements or in adding notes, data, or other information to the notebook.

Additional information concerning documentation is contained in **SOP-4**.

2.5 Decontamination and Investigation Derived Waste

During well redevelopment and sampling, field personnel and equipment will undergo decontamination procedures to ensure the health and safety of those present, to maintain sample integrity, and to minimize the movement of contamination between the work area and off-site locations. Equipment used on-site will be decontaminated prior to beginning work, between sampling locations and/or uses, and prior to demobilizing from the site. Non-disposable purging and sampling equipment will be decontaminated between each sample acquisition by washing with an Alconox® or equivalent detergent wash, a potable water rinse, and a distilled water rinse. Personnel and small equipment decontamination will be performed at the sample locations. Disposable sampling equipment, such as gloves will be collected and bagged on a daily basis and managed in accordance with Solutia procedures. Redevelopment water will be containerized and handled in accordance with Solutia Procedures. Refer to **SOP-6** for decontamination procedures.

3.0 SUPPLEMENTAL SOIL INVESTIGATION

3.1 Lot F

As requested by the USEPA, additional samples will be collected in the vicinity of sample locations S0205, S0206, and S0208 due to PCBs detected in these samples. During previous investigations, PCBs in surface soil [0-2 feet below ground surface (ft bgs)] were detected in Lot F at the above listed sample locations and in nearby soil at the Route 3 Drum Site during the 1986 cleanup. The PCB concentration in exposed surface soil at sample location S0205 was 2.5 mg/kg, which exceeds the TACO Tier 1 criteria for direct contact with soils of 1 mg/kg. A total of four surficial soil samples will be collected from the locations shown in **Figure 3.1** and analyzed for PCBs including:

- One soil sample collected from a depth of 0 to 2 ft bgs located at the midpoint between soil sample locations S0205 and S0206 (new location S0214);
- One soil sample collected from a depth of 0 to 2 ft bgs located at the midpoint between soil sample locations S0205 and S0208; (new location S0215); and
- Two other soil samples collected from a depth of 0 to 2 ft bgs located 100 ft north and 100 ft south of soil sample location S0205 (new locations S0216 and S0217, respectively);

The areal extent of SVOC and lead-containing fill will be assessed in the vicinity of sample location S0110. Sample location S0110 is located in a fill area as indicated from the S0110 boring log and a July 4, 1940 aerial photo. The sample collected from S0110 had detections of 13.2 mg/kg of total PAHs, 1 mg/kg benzo(a)pyrene, and 300 mg/kg lead in exposed surface soil (0-2 ft bgs). The detection for benzo(a)pyrene exceeded direct contact TACO Screening Criteria of 0.8 mg/kg, and the detection of lead approached the TACO Screening Criteria for direct contact. A total of six surficial soil samples will be collected from the locations shown in **Figure 3.1** including:

- Four soil samples collected and analyzed for SVOCs from a depth of 0 to 2 ft bgs located 100 ft north, south, east and west of soil sample location S0110 (new locations S0115-S0118, respectively); and
- Two soil samples collected and analyzed for SVOCs and lead using Method 8270C and 6010B respectively from a depth of 0 to 2 ft bgs 200 ft north and south of soil sample location S0110 (new locations S0119 and S0120, respectively).

The data will be used to help define this fill area and assess the potential human health and ecological risks.

The areal and vertical extent of VOC and SVOC-containing soils that exceed the TACO Tier I criteria for soil to groundwater leaching will be determined in the vicinity of sample location LF-4. According to USEPA, aerial photos indicate past activity (e.g., surface impoundment, disturbed ground) in the area of the LF-series soil sample locations. The LF-series soil sample locations at the southwest corner of Lot F were sampled at 18 to 20 ft bgs and found contain VOCs and SVOCs. LF-4 contained benzene, carbazole, nBnitrosodiphenylamine, and dichloromethane above the TACO Tier 1 soil to groundwater

leaching criteria. A total of four subsurface soil samples will be collected from the locations shown in **Figure 3.1** and analyzed for VOCs and SVOCs including:

- Four soil samples collected and analyzed for VOCs and SVOCs from a depth of 18 to 20 ft bgs located 100 ft north, south, east and west of soil sample location LF-4 (new locations S0121, S0122, S0123 and S0124, respectively.

The samples to be collected are described in **Table 3.1**.

3.2 Former Chlor-Alkali Production Area

As requested by the USEPA, the areal and vertical extent of soils containing mercury at concentrations higher than the TACO Tier I criteria for direct contact with soils or the soil to groundwater leaching criteria will be determined in the area of locations S0916, S0917, S0919, and S0920. A portion of the Former Chlor-Alkali Production Area including samples from soil borings S0916, S0917, S0919, and S0920 exceeds TACO Tier 1 criteria for direct contact with soils for Mercury. The areal extent of this contamination will be defined east of S0916 between S0922 and S0923; west and south of S0917 between S0911 and S0912, and S0910 and S0911; and northwest of S0919 between S0913 and S0914. The deepest sample at 7 to 10 ft bgs obtained at S0919, S0916, and S0920 exceeds the TACO Tier 1 criteria for direct contact with soils for Mercury. Mercury contamination is present in the fill, clayey silt, and silty clay but has not been defined in the deeper sand, which had not been encountered in the borings. A total of 15 subsurface soil samples will be collected from the locations shown in **Figure 3.1** and analyzed for Mercury to define the areal and vertical extent of soils containing mercury at concentrations higher than the TACO Tier I criteria for direct contact with soils or the soil to groundwater leaching criteria including:

- Three soil samples collected from depths of 2 to 3 ft, 6 to 7 ft and 9 to 10 ft bgs at the midpoint between soil sample locations S0910 and S0911 (new location S0925);
- Three soil samples collected from depths of 2 to 3 ft, 6 to 7 ft and 9 to 10 ft bgs at the midpoint between soil sample locations S0911 and S0912 (new location S0926);
- Three soil samples collected from depths of 2 to 3 ft, 6 to 7 ft and 9 to 10 ft bgs at the midpoint between soil sample locations S0913 and S0914 (new location S0927);
- Three soil samples collected from depths of 2 to 3 ft, 6 to 7 ft and 9 to 10 ft bgs at the midpoint between soil sample locations S0922 and S0923 (new location S0928);
- One soil sample collected from a depth of 13 to 15 ft bgs at soil sample location S0916;
- One soil sample collected from a depth of 13 to 15 ft bgs at soil sample location S0919; and
- One soil sample collected from a depth of 13 to 15 ft bgs at soil sample location S0920.

Soils will be sampled and analyzed to confirm whether PCB concentrations are consistently less than the 25 ppm screening criterion in the area of sample locations S0904 and S0905. PCBs in the Former Chlor-Alkali Production Area were detected at 13 and 5 ppm at soil sample locations S0904 and S0905,

respectively within the fill between 9 and 13 ft bgs. A total of nine subsurface soil samples will be collected from the locations shown in **Figure 3.1** and analyzed for PCBs including:

- On soil sample collected from 4 to 6 ft bgs at soil sample location S0902;
- On soil sample collected from 6 to 8 ft bgs at soil sample location S0902;
- On soil sample collected from 2 to 4 ft bgs at soil sample location S0903;
- On soil sample collected from 10 to 12 ft bgs at soil sample location S0907;
- On soil sample collected from 4 to 6 ft bgs at soil sample location S1003;
- On soil sample collected from 3 to 5 ft bgs at soil sample location S1004;
- On fill sample collected from the midpoint between soil sample locations S0904 and S0905 (new location S0929;
- On fill sample collected from the midpoint between soil sample locations S0904 and S0906 (new location S0930; and
- On fill sample collected from the midpoint between soil sample locations S0905 and S0907 (new location S0940).

The samples to be collected are described in **Table 3.1**.

3.3 North Central Plant Process Area

As requested by the USEPA, the presence of VOCs or SVOCs will be determined in the area of S0403. VOCs or SVOCs were not detected between 2 and 4 ft bgs for sample location S0403, although a strong odor and elevated PID reading were noted in the boring log. No intermediate or deep samples were collected from this sample location. Two soil samples will be collected at sampling depths where strong odors and elevated PID readings were noted in the boring log and analyzed for VOCs, SVOCs, Pesticides, Herbicides, and PCBs for sample location S0403 as shown in **Figure 3.1** including:

- One fill/soil sample collected from 1 to 3 ft bgs; and
- One fill/soil sample collected from 10 to 12 ft bgs.

The areal extent of VOCs that exceed either the TACO Tier I criteria for direct contact with soils or the soil to groundwater leaching criteria will be assessed in the area of S0408 and S0409. Aerial photographs indicate that the area including sample locations S0408 and S0409 was a tank farm from at least 1940 through the 1980's. Soil sample locations S0408 and S0409 had elevated chlorobenzene, 1,3-dichloropropene, toluene, ethylbenzene, and xylene (VOC) concentrations at an intermediate depth. A total of four soil samples will be collected from the locations shown in **Figure 3.1** from the intermediate depth with the highest PID reading or most obviously impacted depth between ground surface and 15 ft bgs and analyzed for VOCs including:

- One soil sample collected 100 ft north of soil sample location S0408 (new location S0430);
- One soil sample collected 100 ft northeast of soil sample location S0408 (new location S0432);

- One soil sample collected 100 ft southwest of soil sample location S0408 (new location S0431); and
- One soil sample collected 100 ft west of soil sample location S0408 (new location S0433).

The samples to be collected are described in **Table 3.1**.

3.4 Former Coal Storage Area

As requested by the USEPA, soil samples will be collected to determine the potential risks associated with surface fill in the former coal storage area. The boring logs of soil sample locations S1101, S1102, and S1103 show that fill is present at all three sample locations, varying from 2 to 9 ft bgs. Surface soil samples were not obtained to determine the potential risks associated with surface fill. Therefore, a total of three soil samples will be collected from the locations shown in **Figure 3.1** and analyzed for SVOCs.

- One surface soil collected from 0 to 2 ft bgs at soil sample location S1101;
- One surface soil collected from 0 to 2 ft bgs at soil sample location S1102; and
- One surface soil collected from 0 to 2 ft bgs at soil sample location S1103.

The samples to be collected are described in **Table 3.1**.

3.5 Soil Analytical Parameters

Severn Trent Laboratories will analyze the soil samples for VOCs, SVOCs, Lead, Mercury, PCBs, Pesticides, and Herbicides as specified for each soil sample as described in the above subsections using the following methods:

Parameter	Analytical Method
VOCs	**USEPA Method 8260B**
SVOCs	**USEPA Method 8270C**
Lead	**USEPA Method 6010B**
Mercury	**USEPA Method 7471A**
PCBs	**USEPA Method 8082**
Pesticides	**USEPA Method 8081A**
Herbicides	**USEPA Method 8151A**

Chemical analysis will be conducted in accordance with the Laboratory SOPs located in **Appendix B. Table 3.2** describes the sample container size, preservation and hold times for soil.

3.6 Soil Sampling Procedures

Borings for soil sample collection will be advanced using direct push technology (Geoprobe®). The Geoprobe® hydraulically drives a stainless steel, acetate-lined MacroCore® sampler (2-inch diameter by 4-foot length) to the desired subsurface sample depths. Continuous samples will be collected from the surface to the proposed sampling depths. The subsurface stratigraphy will be logged during drilling operations by a qualified URS field scientist in accordance with the Unified Soil Classification System (USCS) protocols and **SOP-7**. The field scientist will note soil attributes such as color, particle size, consistency, moisture content, structure, plasticity, odor (if obvious) and organic content (if visible). Soil samples from each boring will be visually evaluated for evidence of impact and screened in the field using

a PID. Scaled, color digital photographs will be taken of selected soil samples to provide a record of materials present at this site. At the completion of each soil boring, the boreholes will be backfilled with bentonite chips, and the X-Y coordinates of each location will be obtained. Probing procedures are described in **SOP-8**. PID and sample screening procedures are included in **SOPs 9 and 10**. Samples for VOC analysis will be collected using low-level techniques (**SOP-11**).

To verify field and laboratory procedures, quality assurance/quality control (QA/QC) samples consisting of duplicate samples, and matrix spike/matrix spike duplicate (MS/MSD) samples may be collected and submitted to the laboratory. QA/QC sampling will be taken at a frequency of 10% for duplicates and blanks and 5% for MS/MSDs. Trip blanks will be included with each shipment of samples planned for VOC analysis.

Equipment test checks are to be used to ensure QA/QC for the equipment used in field work.

For proper identification in the field and proper tracking by the analytical laboratory, investigative and QC samples will be labeled in a clear and consistent fashion. Sample labels will be waterproof, or sample containers will be sealed in plastic bags.

A completed sample label will be attached to each investigative or QC sample. The following will be recorded with permanent ink on sample labels by the field sampling team:

- Project name and number,
- Sample number identification,
- Initials of sampler,
- Sampling location (if not already encoded in the sample number),
- Required analysis,
- Date and time of sample collection,
- Space for laboratory sample number, and
- Preservative used, if applicable.

The sample identification system for soil will involve the following nomenclature "S-AA-BB-C-D-EEE" where:

"AA" will denote

- ## - Sample Area

"BB" will denote

- ##- Sample Number

"C" will denote

- #- Initial Sample Depth

"D" will denote

- #- Final Sample Depth

"EEE" will denote QA/QC sample

- DUP- analytical duplicate
- MS or MD – Matrix Spike or Matrix Duplicate

For example, S-04-18-1-2 indicates the soil sample was obtained from sample area 4, the North Production Area, for boring number 18, from 1 to 2 ft bgs.

Samples will be placed on ice inside a cooler immediately following sampling. Sampling containers will be packed in such a way as to help prevent breakage and cross-contamination. Samples will be shipped in coolers, each containing a chain-of-custody form and ice and ice packs to maintain inside temperature at approximately 4°C. Sample coolers will then be sealed between the lid and sides of the cooler with a custody seal prior to shipment. The custody seal will be an adhesive-backed tape that easily rips if it is disturbed. Samples will be shipped to the laboratory by common overnight carrier.

Sample transportation will comply with U.S. Department of Transportation and IICAO/IATA (1999) regulations. Special sampling packing provisions will be made for samples requiring additional protection.

Field personnel will maintain a sampling log sheet containing information sufficient to allow reconstruction of the sample collection and handling procedures at a later time. Chain-of-custody procedures will be instituted and followed throughout the sampling activities. Samples are physical evidence and will be handled according to strict chain-of-custody protocols. The field sampler is personally responsible for the care and custody of the sample until transferred.

Field personnel will record the following information with permanent ink on the chain-of-custody:

- Project identification and number,
- Sample description/location,
- Required analysis,
- Date and time of sample collection,
- Type and matrix of sample,
- Number of sample containers,
- Analysis requested/comments,
- Sampler signature/date/time, and
- Air bill number.

A chain-of-custody document providing all information, signatures, dates, and other information, as required on the example chain-of-custody form included in **SOP-4** will be completed by the field sampler and provided for each sample cooler.

Samples will remain in the custody of the sampler until transfer of custody is completed. Transfer consists

of:

- Delivery of samples to the laboratory sample custodian, and
- Signature of the laboratory sample custodian on the chain-of-custody document as receiving the samples, and signature of sampler, as relinquishing the samples.

If a carrier is used to take samples between the sampler and the laboratory, a copy of the air bill must be attached to the chain-of-custody to maintain proof of custody.

When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the chain-of-custody. The field sampler will sign the chain-of-custody form when relinquishing custody, make a copy to keep with the field logbook, and include the original form in an air-tight plastic bag in the sample cooler with the associated samples.

The laboratory will assign a number for each sample upon receipt. That sample number will be placed on the sample label.

Additional information concerning sample handling, preservation and tracking is contained in **SOPs 4 and 5**.

3.7 Field Documentation

URS personnel will keep a bound field notebook while performing sampling and oversight activities on-site. Forms that will be used include: chain-of-custody, boring log, and soil sampling data sheets and field logbook. The field logbooks will record the following:

- Changes to the plan;
- Personnel conducting the site activities, their arrival and departure times and their destination at the site;
- Location where the work was performed;
- Date, time, weather conditions, equipment, and personnel on site;
- Daily information such as:
  - Work accomplished and the current site status,
  - Equipment calibrations, repairs and results, and
  - Site work zones;
- Specific work activities conducted such as
  - Work zone and headspace readings; and
- Incidents and unusual activities that occur on the site such as, but not limited to, accidents, breaches of security, injuries, equipment failures, or weather related problems.

The following sampling-related information will be recorded in the field logbook by the field sampling team:

- Sample number,
- Project identification,
- Sampling location,
- Required analysis,
- Date and time of sample collection,
- Type and matrix of sample,
- Sampling technique,
- Preservative used, if applicable,
- Sampling conditions,
- Observations,
- Initials of the sampler, and
- Samples collected.

Entries will be signed and dated, and any entry which is to be deleted will have a single cross out which is signed and dated. Photographic records will be developed through the use of digital photographs, showing pre-sampling and post-sampling conditions at each site.

Procedures to evaluate field data for this project primarily include checking for transcription errors on the part of field crew members and review of field notebooks. This task will be the responsibility of the URS Field Leader, who will otherwise not participate in making any of the field measurements or in adding notes, data, or other information to the notebook.

Additional information concerning documentation is contained in **SOP-4**.

3.8 Decontamination and Investigation Derived Waste

The field activities (i.e., sampling, and IDW handling) and data management methods and procedures will follow those discussed in Sections 2.0 and 3.0. Field personnel and equipment will undergo decontamination procedures as described above in Section 2.6. Decontamination fluids will be containerized and handled as discussed in Section 2.6. The IDW including PPE, will be handled as discussed in Section 2.6. Soil cuttings derived from the soil borings will be containerized in 55-gallon, open-top steel drums and managed in accordance with Solutia procedures. The drums will be identified by marking reference information on the lid (e.g., boring number, drum contents, date filled, etc.). Refer to **SOP-6** for decontamination procedures.

4.0 DATA MANAGEMENT, EVALUATION AND REPORTING

4.1 Data Management

The field data and documentation will become a part of the final file. The final file will be the central repository for all documents, which constitute evidence relevant to sampling and analysis activities as described in this plan. URS is the custodian of the file and maintains the contents of files for the site, including all relevant records, logs, field logbooks, pictures, subcontractor reports, data reviews, and the database management system.

The following documentation will supplement the chain-of-custody records:

- Field logbooks and data
- Field collection report
- Photographs and drawings
- Contractor and subcontractor reports
- Correspondence.

The file must be maintained in a secured, limited access area until all submittals for the project have been reviewed and approved, and for a minimum of six years past the submittal date of the final report.

Upon completion of the analyses, URS will begin assimilating the field and laboratory notes. In this way, the file for the samples will be generated. The final file for the samples will be stored at URS and will consist of the following:

- Laboratory data packages, including summary and raw data from the analysis of environmental and QC samples, chromatograms, mass spectra, calibration data, work sheets, and sample preparation notebooks
- Chain-of-custody records
- Data validation reports.

Analytical data will be provided in hard copy and electronic formats. Electronic data will be loaded into a database to facilitate data evaluation and reporting. The data presented in the report will include the data flags provided by STL as well as the qualifiers assigned by URS.

4.2 Data Quality Objectives

The general objective of quality assurance is to collect defensible environmental data of known quality that is adequate for the data's intended use. To accomplish this, data quality objectives (DQOs) were developed. DQOs are qualitative and quantitative statements which clarify the study objective, define the most appropriate types of data to collect, determine the most appropriate conditions from which to collect data, and specify acceptable decisions regarding the data's usage (including reporting limits) to ensure that the collected data will fulfill the project objectives.

Detected analyte concentrations will be compared to relevant Illinois Environmental Protection Agency (IEPA) Tiered Approach to Corrective Action Objectives guidelines. **Tables 4.1** and **4.2** lists the DQOs for

constituents. URS will work with STL to attain the lowest reporting limits to meet the project objectives. However due to technical constraints, achieving reporting limits that are lower than the DQOs might not be feasible for all compounds.

4.3 Quality Assurance Objectives for Measurement

Quality assurance objectives for measurement data are usually expressed in terms of precision, accuracy, completeness, representativeness, and comparability. The investigation will not be considered invalid if these criteria are not fully achieved but variances will trigger QA/QC measures to evaluate, and correct if necessary, any problem areas. The control limits for precision and accuracy as well as detection limits for each laboratory analysis are listed in **Tables 4.3** and **4.4**.

Precision is a measure of the degree to which two or more measurements are in agreement. Field precision is assessed through the collection and measurement of field duplicates at a rate of one duplicate per ten analytical samples. Precision in the laboratory is assessed through the calculation of relative percent differences (RPD) for two or more replicate samples. The equations to be used for precision in this plan are presented in the model QAPP Section 14.2. The precision objective for laboratory analysis will be ± 25 percent RPD for groundwater and ± 50 percent RPD for soil between field duplicates.

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy in the field is assessed through the use of field and trip blanks and through the adherence to all sample handling, preservation, and holding times. Laboratory accuracy is assessed through the analysis of matrix spikes (MS) or laboratory control samples (LCSs), and the determination of percent recoveries. The equation to be used for accuracy in this plan is presented in the model QAPP Section 14.1.

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. Field completeness for this project will be at least 90 percent. Laboratory completeness is a measure of the amount of valid measurements obtained from all the laboratory measurements taken in the project. The equation for completeness is presented in the model QAPP Section 14.3. Laboratory completeness for this project will be at least 95 percent. Results assigned an "R" qualifier would be unusable.

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the plan is followed and that proper sampling techniques are used. Representativeness in the laboratory data is ensured by using the proper analytical procedures, meeting sample holding times, and analyzing and assessing the field duplicate samples and matrix spike duplicate samples. The sampling network is designed to provide data representative of site conditions. During development of this network, consideration is given to existing analytical data, past site practices, and physical setting and processes.

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability can be related to precision and accuracy since these quantities are measures of data

reliability. Samples from the same media are considered comparable if the procedures for collecting the samples are complied with and if the units of measurement are the same. Comparability is assured through the use of a laboratory for this project that uses established and approved analytical methods, protocols, and a laboratory quality control program designed to establish consistency in the performance of the analytical process. All data will be subjected to strict QA/QC procedures and reported in a consistent manner to allow for comparison across data sets.

4.4 Data Validation

Data validation will be performed by the URS QA Manager in accordance with QA/QC criteria established in the USEPA Region 5 Model QAPP. Excursions from QA/QC criteria will be qualified based on guidance provided in the following documents where applicable to the reference methods:

- USEPA Contract Laboratory Program National Functional Guidelines for Low Concentration Organic Data Review. USEPA 540/R-94/012 (USEPA, 2001)
- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. USEPA 540/R-94/013 (USEPA, 2004)

A Level III validation will be performed for all data, and a Level IV validation will be performed for approximately 10 percent of the data. Data validators will recalculate approximately 10 percent of the laboratory sample calculations using raw data when verifying sample results. In addition, data validators will review approximately 10 percent of the raw data to verify that compound identification was performed correctly and transcription errors are not present.

Data quality will be evaluated using laboratory or method control limits. Any control limits outside of the acceptable range shall be identified and reported. Sample data will be qualified based on excursions from laboratory or method control limits. Then, data validators will check corrective actions and results of reanalysis and document these events in the validation report.

Minor deficiencies in the data generation process noted in the data validation will result in approximation of sample data. Approximation of a data point indicates uncertainty in the reported concentration of the chemical but not its assigned identity. Major deficiencies noted in the data, validation will result in the rejection of sample results. Rejected data would be considered unusable for quantitative or qualitative purposes. Data qualifiers may include the following:

U Indicates that the compound was analyzed for, but was not detected. The sample quantitation limit is presented and adjusted for dilution and percent moisture. This qualifier is also used to signify that the detection limit of an analyte was raised as a result of analytes detected in laboratory and/or field blank samples.

J Indicates that the detected sample result should be considered approximate based on excursions from QA/QC criteria.

UJ Indicates that the detection limit for the analyte in this sample should be considered approximate based on excursions from QA/QC criteria.

R Indicates that the previously reported detection limit or sample result has been rejected due to a

major excursion from QA/QC criteria, for example percent recoveries of less than ten percent. The data should not be used for qualitative or quantitative purposes.

The following method specific QA/QC parameters will be evaluated (at a minimum) during the data validation, where applicable.

Analyses for VOCs and SVOCs (where applicable)

Holding times, sample preservation, and percent solids

Dilutions

GC/MS instrument performance (Level IV validation only)

Initial and continuing calibration (Level IV validation only)

Blank analysis

Surrogate recovery

MS/MSD analysis

Field duplicate analysis

Laboratory Control Sample (LCS) analysis

Internal standards performance

Compound identification and quantification (Level IV validation only)

Documentation completeness

Overall assessment.

Analyses for Pesticides and PCBs, (where applicable):

Holding times, sample preservation, and percent solids

Dilutions

Initial and continuing calibration (Level IV validation only)

Blank analysis

Surrogate recovery

MS/MSD analysis

Field duplicate analysis

Laboratory Control Sample (LCS) analysis

Compound identification and quantification (Level IV validation only)

Documentation completeness

Overall assessment.

Analyses for Herbicides, (where applicable):

- Holding times, sample preservation, and percent solids
- Dilutions
- Initial and continuing calibration (Level IV validation only)
- Blank analysis
- Surrogate recovery
- MS/MSD analysis
- Field duplicate analysis
- Laboratory Control Sample (LCS) analysis
- Compound identification and quantification (Level IV validation only)
- Documentation completeness
- Overall assessment.

Analyses for Metals, (where applicable):

- Holding times, sample preservation, and percent solids
- Dilutions
- Calibration (Level IV validation only)
- Blank analysis
- Spike analysis
- Field duplicate analysis
- Laboratory Control Sample (LCS) analysis
- Sample Result Verification (Level IV validation only)
- ICP Interference Check Samples (Level IV validation only)
- Laboratory Duplicates
- ICP Serial Dilution
- Documentation completeness
- Overall assessment.

Analyses for Mercury, (where applicable):

- Holding times and sample preservation
- Dilutions
- Initial and continuing calibration (Level IV validation only)

Compound identification and quantification

Blank analysis

Spike analysis

Field duplicate analysis

Laboratory Control Sample (LCS) analysis

Laboratory Duplicates

Documentation completeness

Overall assessment.

The data will be provided to URS in an electronic format that will be uploaded into a database to facilitate data review and evaluation. The report tables will be generated from the database. These tables will include the data flags provided by Savannah Labs as well as the data validation qualifiers assigned by URS.

Laboratory provided results (Form 1's) will also be included in the final report with the transcribed data qualifiers.

4.5 Reporting

The Supplemental Soil and Groundwater Sampling Report will be prepared and submitted to USEPA after sampling, sample analysis and data validation are completed. The report will include a groundwater-elevation contour map, a summary of the laboratory analytical data (soil and groundwater), and a discussion of the following objectives requested by the USEPA in a letter dated May 4, 2005.

The report will provide the following types of information for the Lot F Drum: the general procedures for and frequency of inspections and maintenance of the cap, a list of activities and costs of inspection and maintenance included in the final corrective measures array analysis and a figure delineating the boundaries of the Route 3 Drum Site and location of each monitoring well sampled with individual constituent concentrations found in groundwater at each monitoring well sampled. The results of this work will be used to evaluate if the interim actions conducted at the Lot F drum Site in addition to groundwater collection at the Sauget Area 2 Groundwater Migration Control System, is sufficient to protect human health and the environment.

The report will define the vertical and areal extent of contamination at Lot F, the Former Chlor-Alkali Production Area, the North Central Plant Process Area, and the Former Coal Storage Area and address the following specific requests.

<u>Lot F:</u>

- The areal extent of PCB-containing soils and the associated human health and ecologic risk,
- the extent of the fill area,
- and the areal and vertical extent of VOC and SVOC-containing soils that exceed the TACO Tier I criteria for soil to groundwater leaching in Lot F.

Former Chlor-Alkali Production Area:

- The areal and vertical extent of soils containing mercury at concentrations higher than the TACO Tier I criteria for direct contact with soils or the soil to groundwater leaching criteria,
- and a confirmation that PCB concentrations are consistently less than the 25 ppm screening criteria.

North Central Process Area:

- The results of the two soil samples collected for sample location S0403 to determine if the elevated PID readings indicated the presence of VOCs,
- and the areal and vertical extent of VOCs that exceed either the TACO Tier I criteria for direct contact with soils or the soil to groundwater leaching criteria.

Former Coal Storage Area:

- The results and analysis of three soil samples collected to determine the potential risks of the contaminants present in the fill.

5.0 SCHEDULE

The proposed schedule for the supplemental soil and groundwater sampling work to be completed is as follows:

Task	July	August	September
Work Plan Approved			
Field Sampling Activities			
Laboratory Analysis			
Data Review and Validation			
Report Compilation			

6.0 REFERENCES

U.S. Environmental Protection Agency (USEPA) Region 5, 1998, *RCRA QAPP Instructions*, April 1998, Chicago, IL.

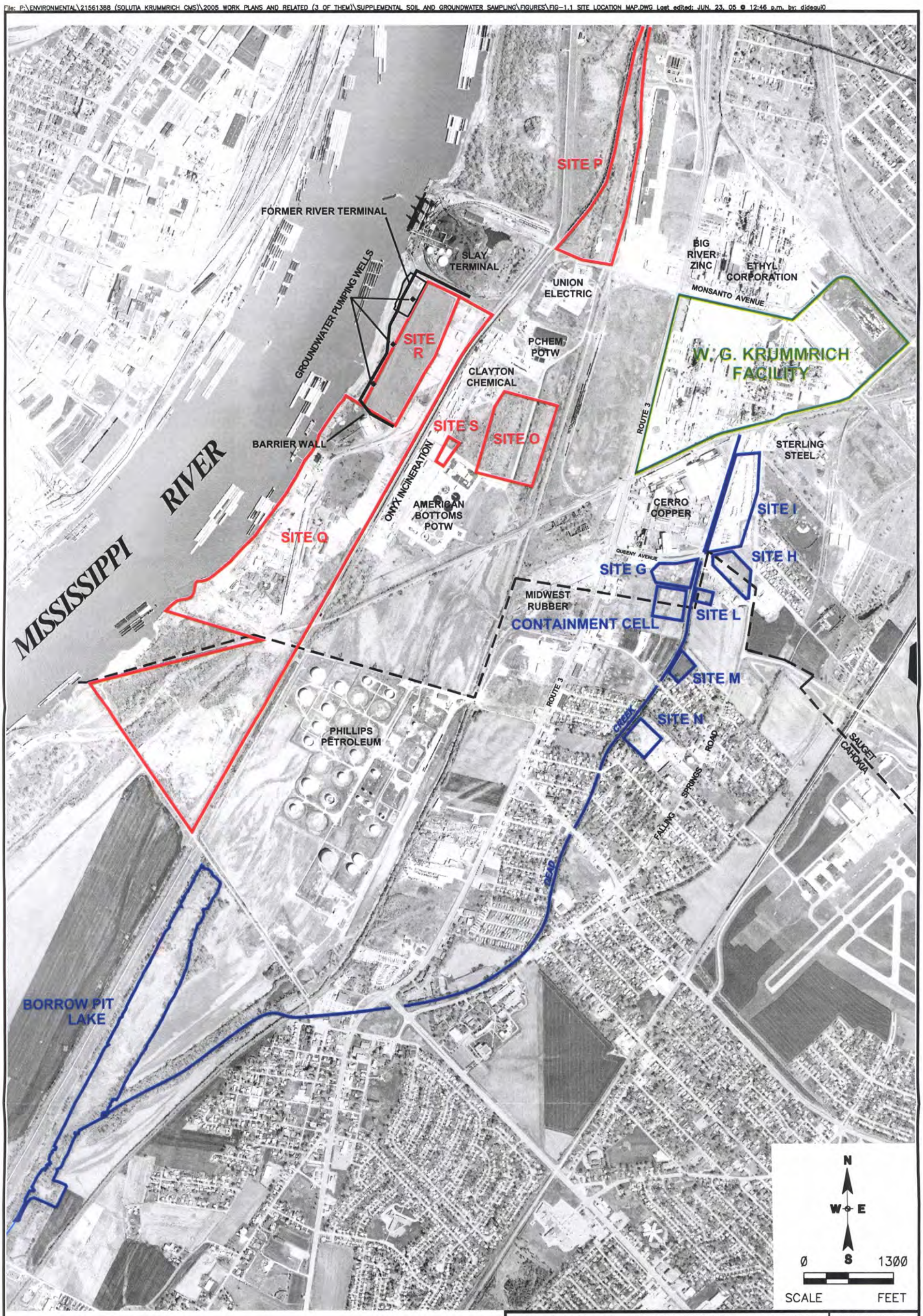
URS Corporation, 2004. *RCRA Corrective Measures Study (CMS), Solutia Inc., W.G. Krummrich Facility, Sauget, Illinois.*

URS Corporation, 2005. *RCRA Corrective Measures Study (CMS), Response to Comments, Solutia Inc., W.G. Krummrich Facility, Sauget, Illinois.*

URS Corporation, 2005. *RCRA Corrective Measures Study, Response to May 4, 2005 USEPA Comments, Solutia Inc.., W.G. Krummrich Facility Investigation, Sauget, Illinois.*

FIGURES

File: P:\ENVIRONMENTAL\21561388 (SOLUTIA KRUMMRICH CMS)\2005 WORK PLANS AND RELATED (3 OF THEM)\SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING\FIGURES\FIG-1.1 SITE LOCATION MAP.DWG Last edited: JUN. 23, 05 @ 12:46 p.m. by: dideguil0



LEGEND

- W.G. KRUMMRICH FACILITY
- SAUGET AREA #1
- SAUGET AREA #2

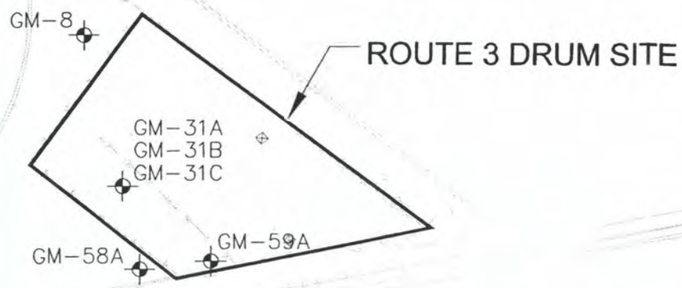
SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING PLAN W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS	PROJECT NO. 21561573.00000
URS	
DRN. BY: 2/9/05 DSGN. BY: tja CHKD. BY:	Site Location Map
	FIG. NO. 1.1

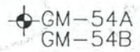
SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING PLAN W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS	PROJECT NO. 21561573.00000

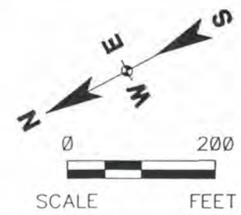
URS

DRN. BY: 4/9/05 DSGN. BY: cm CHKD. BY:	Facility Location Map	FIG. NO. 1.2

LOT F





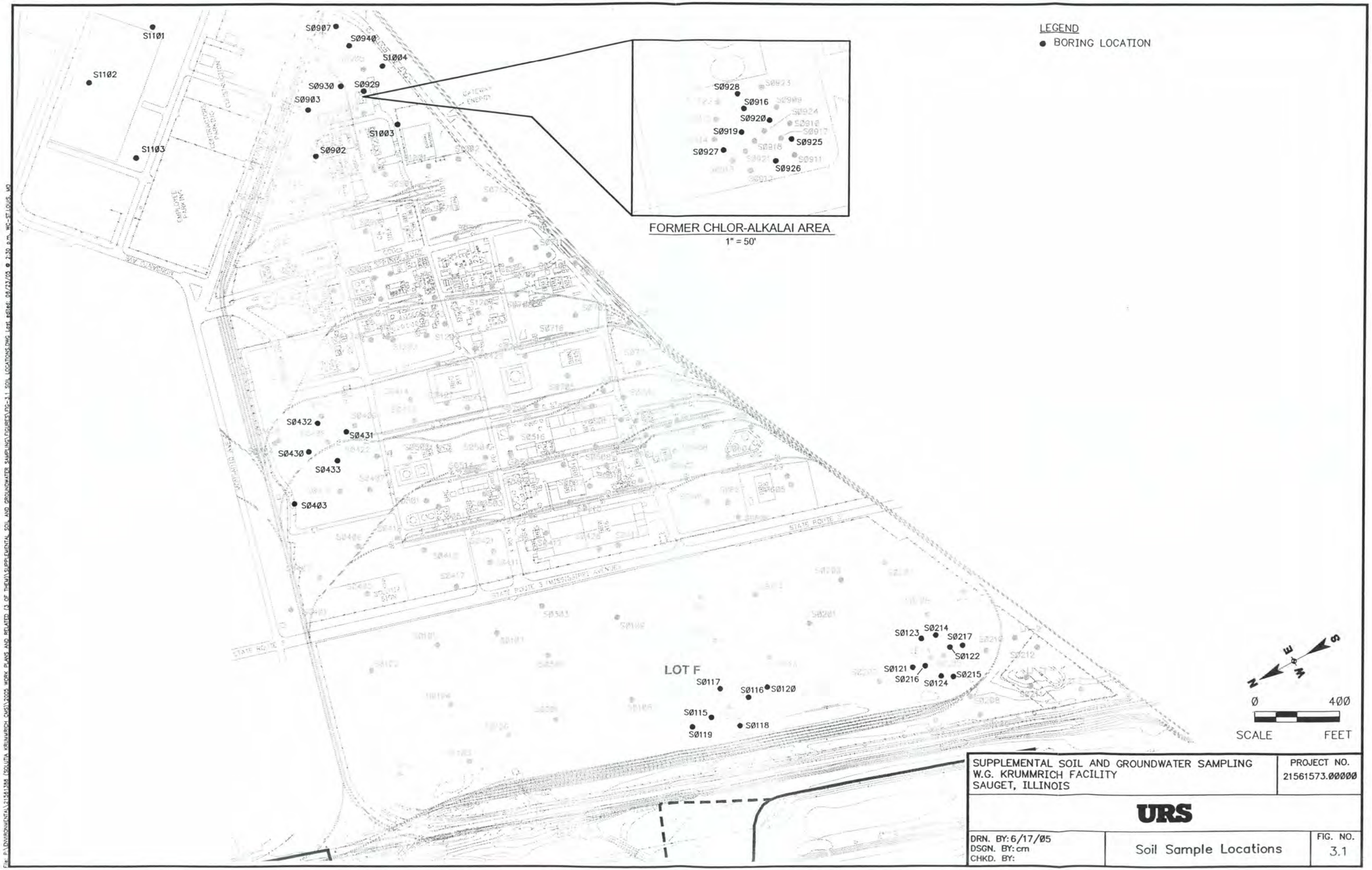


LEGEND

MONITORING WELL LOCATION

SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING PLAN W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS		PROJECT NO. 21561573.00000
URS		
DRN. BY: djd 2/10/05 DSGN. BY: bh CHKD. BY:	Monitoring Well Locations	FIG. NO. 2.1

File: P:\ENVIRONMENTAL\21561388 (SOLUTIA KRUMMRICH CMS)\2005 WORK PLANS AND RELATED (3 OF THEM)\SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING\FIGURES\FIG-2.1 MW LOCATIONS.DWG Last edited: 06/23/05 @ 4:17 p.m. WC-ST.LOUIS, MO



SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS	PROJECT NO. 21561573.00000
URS	
DRN. BY: 6/17/05 DSGN. BY: cm CHKD. BY:	Soil Sample Locations
	FIG. NO. 3.1

TABLE 2.1
MONITORING WELL COMPLETION SUMMARY
ROUTE 3 DRUM SITE

Monitoring Well Identification	Top of Casing Elevation (ft MSL)	Total Well Depth (ft btoc)	Bottom of Well Elevation (ft MSL)	Screened Interval (ft btoc)	Screened Interval Elevation (ft MSL)
GM-31A	418.63	33.15	385.48	(19.00-39.00)	(399.63-379.63)
GM-31B	418.92	86.21	332.71	(65.50-85.50)	(353.42-333.42)
GM-31C	419.29	119.4	299.89	(97.00-117.00)	(322.29-302.29)
GM-54A	404.93	40.75	364.18	(66.50-86.50)	(338.43-318.43)
GM-54B	405.11	92.15	312.96	(66.50-86.50)	(338.61-318.61)
GM-58A	414.24	41.8	372.44	(19.40-39.40)	(394.84-374.84)
GM-59A	413.57	41.53	372.04	(19.00-39.00)	(394.57-374.57)
GM-8	418.49	35.76	382.73	(19.00-34.00)	(399.49-384.49)

Notes:

1) MSL=Mean Sea Level

2) btoc= below top of casing

3) The total well depths for GM-54A, GM-54B, and GM-58A shown on the table were measured between January 24, 2000 and February 1, 2000.

4) The total well depths for GM-31A, GM-31B, GM-31C, GM-59A, and GM-8 shown on the table were measured June 15, 2005.

Table 2.2 Recommended Sample Containers, Preservation, and Hold Times for Parameters Measured in Wastewater and Groundwater Matrices

Semivolatiles by GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Base/Neutrals/Acids	8270	2 x 1-L Amb G	None (6)	7 days/40 days

PCBs by GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Polychlorinated Byphenols	8082	1-L Amb G	None (6)	7 days/40 days

Table 3.1
Soil Sample Description

Sample Location ID	Area	Location Description	Sample Depth (ft bgs)	Sample Parameters	EPA Method
S0214	Lot F	Midpoint between S0205 and S0206	0-2	PCBs	USEPA Method 680
S0215	Lot F	Midpoint between S0205 and S0208	0-2	PCBs	USEPA Method 680
S0216	Lot F	100' north of S0205	0-2	PCBs	USEPA Method 680
S0217	Lot F	100' south of S0205	0-2	PCBs	USEPA Method 680
S0115	Lot F	100' north of S0110	0-2	SVOCs	USEPA Method 8270C
S0116	Lot F	100' south of S0110	0-2	SVOCs	USEPA Method 8270C
S0117	Lot F	100' east of S0110	0-2	SVOCs	USEPA Method 8270C
S0118	Lot F	100' west of S0110	0-2	SVOCs	USEPA Method 8270C
S0119	Lot F	200' north of S0110	0-2	SVOCs Lead	USEPA Method 8270C USEPA Method 6010B
S0120	Lot F	200' south of S0110	0-2	SVOCs Lead	USEPA Method 8270C USEPA Method 6010B
S0121	Lot F	100' north of LF-4	18-20	VOCs SVOCs	USEPA Method 8260B USEPA Method 8270C
S0122	Lot F	100' south of LF-4	18-20	VOCs SVOCs	USEPA Method 8260B USEPA Method 8270C
S0123	Lot F	100' east of LF-4	18-20	VOCs SVOCs	USEPA Method 8260B USEPA Method 8270C
S0124	Lot F	100' west of LF-4	18-20	VOCs SVOCs	USEPA Method 8260B USEPA Method 8270C
S0925	Former Chlor-Alkali Production Area	Midpoint between S0910 and S0911	2-3 6-7 9-10	Mercury	USEPA Method 7470C
S0926	Former Chlor-Alkali Production Area	Midpoint between S0911 and S0912	2-3 6-7 9-10	Mercury	USEPA Method 7470C

Table 3.1
Soil Sample Description

Sample Location ID	Area	Location Description	Sample Depth (ft bgs)	Sample Parameters	EPA Method
S0927	Former Chlor-Alkali Production Area	Midpoint between S0913 and S0914	2-3 6-7 9-10	Mercury	USEPA Method 7470C
S0928	Former Chlor-Alkali Production Area	Midpoint between S0922 and S0923	2-3 6-7 9-10	Mercury	USEPA Method 7470C
S0916	Former Chlor-Alkali Production Area	S0916	13-15	Mercury	USEPA Method 7470C
S0919	Former Chlor-Alkali Production Area	S0919	13-15	Mercury	USEPA Method 7470C
S0920	Former Chlor-Alkali Production Area	S0920	13-15	Mercury	USEPA Method 7470C
S0902	Former Chlor-Alkali Production Area	S0902	4-6 6-8	PCBs	USEPA Method 680
S0903	Former Chlor-Alkali Production Area	S0903	2-4	PCBs	USEPA Method 680
S0907	Former Chlor-Alkali Production Area	S0907	10-12	PCBs	USEPA Method 680
S1003	Former Chlor-Alkali Production Area	S1003	4-6	PCBs	USEPA Method 680
S1004	Former Chlor-Alkali Production Area	S1004	3-5	PCBs	USEPA Method 680
S0929	Former Chlor-Alkali Production Area	Midpoint between S0904 and S0905	Fill sample	PCBs	USEPA Method 680
S0930	Former Chlor-Alkali Production Area	Midpoint between S0904 and S0906	Fill sample	PCBs	USEPA Method 680

Table 3.1
Soil Sample Description

Sample Location ID	Area	Location Description	Sample Depth (ft bgs)	Sample Parameters	EPA Method
S0940	Former Chlor-Alkali Production Area	Midpoint between S0905 and S0907	Fill sample	PCBs	USEPA Method 680
S0403	North Central Plant Process Area	S0403	1-3 10-12	VOCs SVOCs Pesticides Herbicides PCBs	USEPA Method 8260B USEPA Method 8270C USEPA Method 8081A USEPA Method 8151A USEPA Method 680
S0430	North Central Plant Process Area	100' north of S0408	Highest PID Reading 0-15	VOCs	USEPA Method 8260B
S0431	North Central Plant Process Area	100' south of S0408	Highest PID Reading 0-15	VOCs	USEPA Method 8260B
S0432	North Central Plant Process Area	100' east of S0408	Highest PID Reading 0-15	VOCs	USEPA Method 8260B
S0433	North Central Plant Process Area	100' west of S0408	Highest PID Reading 0-15	VOCs	USEPA Method 8260B
S1101	Former Coal Storage Area	S1101	0-2	SVOCs	USEPA Method 8270C
S1102	Former Coal Storage Area	S1102	0-2	SVOCs	USEPA Method 8270C
S1103	Former Coal Storage Area	S1103	0-2	SVOCs	USEPA Method 8270C

Table 3.2 Recommended Sample Containers, Preservation, and Hold Time for Parameters Measured in Soil and Solid Matrices

General Chemistry

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Organic Carbon, Total (TOC)	Lloyd Kahn (Combustion)	250-ml P	None	28 days

Volatile Organics – GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Volatiles	8260(5035)	3 x 5-g Encore plus125-ml G Amb	5ml 5% sodium bisulfate solution, methanol, or frozen in water	14 days

Semivolatiles by GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Base/Neutrals/Acids	8270	250-ml or 500-ml amb G	None	14 days/40 days

Organochloride Pesticides and PCBs by GC

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Polychlorinated Byphenols	8082	500-ml amb G	None	14 days/40 days

Chlorinated Herbicides

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Herbicides	8151A	500-ml amb G	None (6)	14 days/40 days

Metals

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Metals (except Mercury)	ICP: 6010 GFAA:7000-series	250 ml P	None	6 months

Metals

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Mercury	7471A	250 ml P or G	None	28 days

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Polychlorinated Biphenyls as Aroclors by GC/EC							
PCB 1016 (MS)	12674-11-2	8082	0.14	1.0	NA	Not Available	NA
PCB 1221	11104-28-2	8082	0.50	2.0	NA	Not Available	NA
PCB 1232	11141-16-5	8082	0.22	1.0	NA	Not Available	NA
PCB 1242	53469-21-9	8082	0.22	1.0	NA	Not Available	NA
PCB 1248	12672-29-6	8082	0.33	1.0	NA	Not Available	NA
PCB 1254	11097-69-1	8082	0.20	1.0	NA	Not Available	NA
PCB 1260 (MS)	11096-82-5	8082	0.17	1.0	NA	Not Available	NA
PCB 1268	11100-14-4	8082	0.10	1.0	NA	Not Available	NA
Surrogates		8082					
Decachlorobiphenyl	2051-24-3	8082	NA	NA	NA	Not Available	NA
2,4,5,6-Tetrachloro-m-xylene	877-09-8	8082	NA	NA	NA	Not Available	NA
Semivolatiles in Groundwater by GC/MS							
1,2,4,5-Tetrachlorobenzene	95-94-3	8270 (3520)	1.2	10.00	NA	Not Available	NA
1,2,4-Trichlorobenzene (MS)	120-82-1	8270 (3520)	0.54	10.00	70.00	IEPA TACO Class I	Yes
1,2-Dichlorobenzene	95-50-1	8270 (3520)	1.0	10.00	600.00	IEPA TACO Class I	Yes
1,2-Diphenyl hydrazine	122-66-7	8270 (3520)	0.97	10.00	NA	Not Available	NA
1,3,5-Trinitrobenzene	99-35-4	8270 (3520)	0.62	10.00	210.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	Yes
1,3-Dichlorobenzene	541-73-1	8270 (3520)	0.55	10.00	5.50	Region 9 (Tap Water)	No
1,4-Dichlorobenzene(MS)	106-46-7	8270 (3520)	0.52	10.00	75.00	IEPA TACO Class I	Yes
1,4-Dioxane	123-91-1	8270 (3520)	2.3	10.00	1.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	No
1,4-Naphthoquinone	130-15-4	8270 (3520)	0.67	10.00	NA	Not Available	NA
1-Methylnaphthalene	90-12-0	8270 (3520)	0.62	10.00	28.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	Yes
1-Naphthylamine	134-32-7	8270 (3520)	1.2	10.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
2,3,4,6-Tetrachlorophenol	58-90-2	8270 (3520)	0.47	10.00	NA	Not Available	NA
2,4,5-Trichlorophenol	95-95-4	8270 (3520)	0.63	10.00	700.00	IEPA TACO Class I	Yes
2,4,6-Trichlorophenol	88-06-2	8270 (3520)	0.70	10.00	10.00	IEPA TACO Class I	Yes
2,4-Dichlorophenol	120-83-2	8270 (3520)	0.66	10.00	21.00	IEPA TACO Class I	Yes
2,4-Dimethylphenol	105-67-9	8270 (3520)	1.0	10.00	140.00	IEPA TACO Class I	Yes
2,4-Dinitrophenol	51-28-5	8270 (3520)	5.0	50.00	14.00	IEPA TACO Class I	No
2,4-Dinitrotoluene (MS)	121-14-2	8270 (3520)	0.56	10.00	0.02	IEPA TACO Class I	No
2,6-Dichlorophenol	87-65-0	8270 (3520)	0.69	10.00	NA	Not Available	NA
2,6-Dinitrotoluene	606-20-2	8270 (3520)	0.57	10.00	0.31	IEPA TACO Class I	No
2-Acetylaminofluorene	53-96-3	8270 (3520)	1.4	10.00	NA	Not Available	NA
2-Chloronaphthalene	91-58-7	8270 (3520)	0.62	10.00	NA	Not Available	NA
2-Chlorophenol (MS)	95-57-8	8270 (3520)	0.72	10.00	35.00	IEPA TACO Class I	Yes
2-Methyl phenol	95-48-7	8270 (3520)	0.70	10.00	350.00	IEPA TACO Class I	Yes
2-Methylnaphthalene	91-57-6	8270 (3520)	0.53	10.00	140.00	IEPA TACO Class I - Surrogate Chemical: Napthalene (per USEPA National Center for Environmental Assessment)	Yes
2-Naphthylamine	91-59-8	8270 (3520)	0.64	10.00	NA	Not Available	NA
2-Nitroaniline	88-74-4	8270 (3520)	0.69	50.00	1.00	Region 9 (Tap Water)	No
2-Nitrophenol	88-75-5	8270 (3520)	0.73	10.00	NA	Not Available	NA
2-Picoline	109-06-8	8270 (3520)	0.87	10.00	7.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	No
3- and 4-Methyl phenol	106-44-5	8270 (3520)	0.66	10.00	35.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	Yes
3,3'-Dichlorobenzidine	91-94-1	8270 (3520)	1.0	20.00	20.00	IEPA TACO Class I	Yes
3,3'-Dimethylbenzidine	119-93-7	8270 (3520)	2.5	20.00	NA	Not Available	NA
3-Methylcholanthrene	56-49-5	8270 (3520)	1.4	10.00	NA	Not Available	NA
3-Nitroaniline	99-09-2	8270 (3520)	0.69	50.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
4,6-Dinitro-2-methylphenol	534-52-1	8270 (3520)	1.0	50.00	0.01	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
4-Aminobiphenyl	92-67-1	8270 (3520)	0.68	10.00	NA	Not Available	NA
4-Bromophenyl phenyl ether	101-55-3	8270 (3520)	0.85	10.00	NA	Not Available	NA
4-Chloro-3-methyl-phenol (MS)	59-50-7	8270 (3520)	0.55	10.00	NA	Not Available	NA
4-Chloroaniline	106-47-8	8270 (3520)	0.52	20.00	28.00	IEPA TACO Class I	Yes
4-Chlorophenylphenyl ether	7005-72-3	8270 (3520)	0.56	10.00	NA	Not Available	NA
4-Nitroaniline	100-01-6	8270 (3520)	0.85	50.00	NA	Not Available	NA
4-Nitrophenol (MS)	100-02-7	8270 (3520)	3.4	50.00	290.00	Region 6 (Tap Water)	Yes
4-Nitroquinoline-1-oxide	56-57-5	8270 (3520)	5.0	20.00	NA	Not Available	NA
5-Nitro-o-toluidine	99-55-8	8270 (3520)	1.2	10.00	NA	Not Available	NA
7,12-Dimethylbenz(a)anthracene	57-97-6	8270 (3520)	1.3	10.00	NA	Not Available	NA
a,a-Dimethylphenethylamine	122-09-8	8270 (3520)	10	2000.00	NA	Not Available	NA
Acenaphthene (MS)	83-32-9	8270 (3520)	0.64	10.00	420.00	IEPA TACO Class I	Yes
Acenaphthylene	208-96-8	8270 (3520)	0.58	10.00	NA	Not Available	NA
Acetophenone	98-86-2	8270 (3520)	0.83	10.00	NA	Not Available	NA
Aniline	62-53-3	8270 (3520)	0.65	20.00	23.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Anthracene	120-12-7	8270 (3520)	0.88	10.00	2100.00	IEPA TACO Class I	Yes
Aramite	140-57-8	8270 (3520)	1.7	10.00	NA	Not Available	NA
Benzidine	92-87-5	8270 (3520)	14	80.00	NA	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Benzo(a)anthracene	56-55-3	8270 (3520)	0.58	10.00	0.13	IEPA TACO Class I	No
Benzo(a)pyrene	50-32-8	8270 (3520)	1.0	10.00	0.20	IEPA TACO Class I	No
Benzo(b)fluoranthene	205-99-2	8270 (3520)	0.78	10.00	0.18	IEPA TACO Class I	No

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Benzo(g,h,i)perylene	191-24-2	8270 (3520)	1.0	10.00	0.21	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Benzo(k)fluoranthene	207-08-9	8270 (3520)	0.73	10.00	0.17	IEPA TACO Class I	No
Benzoic acid	65-85-0	8270 (3520)	2.5	50.00	28000.00	IEPA TACO Class I	Yes
Benzyl alcohol	100-51-6	8270 (3520)	0.80	10.00	2100.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Bis(2-chloroethoxy) methane	111-91-1	8270 (3520)	0.78	10.00	NA	Not Available	NA
Bis(2-chloroethyl) ether	111-44-4	8270 (3520)	0.72	10.00	10.00	IEPA TACO Class I	Yes
Bis(2-chloroisopropyl)ether (2,2-Oxybis(1-chloropropane))	111-44-4	8270 (3520)	0.82	10.00	10.00	IEPA TACO Class I	Yes
Bis(2-ethylhexyl) phthalate	117-81-7	8270 (3520)	0.98	10.00	6.00	IEPA TACO Class I	No
Butyl benzyl phthalate	85-68-7	8270 (3520)	0.70	10.00	1400.00	IEPA TACO Class I	Yes
Carbazole	86-74-8	8270 (3520)	0.82	10.00	3.40	Region 9 (Tap Water)	No
Chrysene	218-01-9	8270 (3520)	1.0	10.00	1.50	IEPA TACO Class I	No
Diallate	2303-16-4	8270 (3520)	1.6	10.00	NA	Not Available	NA
Dibenz(a,h)anthracene	53-70-3	8270 (3520)	1.0	10.00	0.30	IEPA TACO Class I	No
Dibenzofuran	132-64-9	8270 (3520)	0.69	10.00	28.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Diethyl phthalate	84-66-2	8270 (3520)	0.59	10.00	5600.00	IEPA TACO Class I	Yes
Dimethoate	60-51-5	8270 (3520)	0.64	10.00	NA	Not Available	NA
Dimethylphthalate	131-11-3	8270 (3520)	0.59	10.00	NA	Not Available	NA
Di-n-butyl phthalate	84-74-2	8270 (3520)	0.82	10.00	4000.00	IEPA TACO Class I	Yes
Di-n-octyl phthalate	117-84-0	8270 (3520)	0.80	10.00	140.00	IEPA TACO Class I	Yes
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	88-85-7	8270 (3520)	2.5	10.00	7.00	IEPA TACO Class I	No

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Diphenylamine/ Nitrosodiphenylamine	122-39-4	8270 (3520)	0.80	10.00	175.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Disulfoton	298-04-4	8270 (3520)	0.78	10.00	0.28	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Ethyl methanesulfonate	62-50-0	8270 (3520)	0.90	10.00	NA	Not Available	NA
Ethyl parathion	56-38-2	8270 (3520)	0.89	10.00	168.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Famphur	52-85-7	8270 (3520)	2.2	10.00	NA	Not Available	NA
Fluoranthene	206-44-0	8270 (3520)	1.0	10.00	280.00	IEPA TACO Class I	Yes
Fluorene	86-73-7	8270 (3520)	0.55	10.00	280.00	IEPA TACO Class I	Yes
Hexachlorobenzene	118-74-1	8270 (3520)	1.1	10.00	0.06	IEPA TACO Class I	No
Hexachlorobutadiene	87-68-3	8270 (3520)	1.0	10.00	1.40	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Hexachlorocyclopentadiene	77-47-4	8270 (3520)	5.0	10.00	50.00	IEPA TACO Class I	Yes
Hexachloroethane	67-72-1	8270 (3520)	1.0	10.00	7.00	IEPA TACO Class I	No
Hexachlorophene	70-30-4	8270 (3520)	47	5000.00	NA	Not Available	NA
Hexachloropropene	1888-71-7	8270 (3520)	1.2	10.00	NA	Not Available	NA
Indeno(1,2,3-cd)pyrene	193-39-5	8270 (3520)	0.62	10.00	0.43	IEPA TACO Class I	No
Isophorone	78-59-1	8270 (3520)	0.67	10.00	1400.00	IEPA TACO Class I	Yes
Isosafrole	120-58-1	8270 (3520)	0.96	10.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
m-Dinitrobenzene	99-65-0	8270 (3520)	0.68	10.00	0.70	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Methapyrilene	91-80-5	8270 (3520)	1.9	2000.00	NA	Not Available	NA
Methyl parathion	298-00-0	8270 (3520)	0.78	10.00	NA	Not Available	NA
Methylmethanesulfonate	66-27-3	8270 (3520)	0.85	10.00	NA	Not Available	NA
Naphthalene	91-20-3	8270 (3520)	0.76	10.00	140.00	IEPA TACO Class I	Yes
Nitrobenzene	98-95-3	8270 (3520)	0.93	10.00	3.50	IEPA TACO Class I	No
Nitrosodiphenylamine/ Diphenylamine	86-30-6, 122-39-4	8270 (3520)	0.80	10.00	175.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
N-Nitrosodiethylamine	55-18-5	8270 (3520)	0.82	10.00	NA	Not Available	NA
N-Nitrosodimethylamine	62-75-9	8270 (3520)	1.1	10.00	NA	Not Available	NA
N-Nitrosodi-n-butylamine	924-16-3	8270 (3520)	0.78	10.00	NA	Not Available	NA
N-Nitrosodi-n-propylamine(MS)	621-64-7	8270 (3520)	0.62	10.00	1.80	IEPA TACO Class I	No
N-Nitrosomethylethylamine	10595-95-6	8270 (3520)	2.6	10.00	NA	Not Available	NA
N-Nitrosomorpholine	59-89-2	8270 (3520)	1.1	10.00	NA	Not Available	NA
N-Nitrosopiperidine	100-75-4	8270 (3520)	1.2	10.00	NA	Not Available	NA
N-Nitrosopyrrolidine	930-55-2	8270 (3520)	0.57	10.00	NA	Not Available	NA
o,o,o-Triethyl-phosphorothioate	126-68-1	8270 (3520)	1.3	10.00	NA	Not Available	NA
o-Toluidine	95-53-4	8270 (3520)	0.58	10.00	NA	Not Available	NA
p-(Dimethylamino)azobenzene	60-11-7	8270 (3520)	1.2	10.00	NA	Not Available	NA
Pentachlorobenzene	608-93-5	8270 (3520)	1.0	10.00	NA	Not Available	NA
Pentachloronitrobenzene	82-68-8	8270 (3520)	1.2	10.00	21.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Pentachlorophenol (MS)	87-86-5	8270 (3520)	2.5	50.00	1.00	IEPA TACO Class I	No
Phenacetin	62-44-2	8270 (3520)	0.82	10.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Phenanthrene	85-01-8	8270 (3520)	0.74	10.00	0.21	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Phenol (MS)	108-95-2	8270 (3520)	0.99	10.00	100.00	IEPA TACO Class I	Yes
Phorate	298-02-2	8270 (3520)	0.83	10.00	14.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
p-Phenylenediamine	106-50-3	8270 (3520)	37	2000.00	NA	Not Available	NA
Pronamide	23950-58-5	8270 (3520)	0.72	10.00	NA	Not Available	NA
Pyrene(MS)	129-00-0	8270 (3520)	1.0	10.00	210.00	IEPA TACO Class I	Yes
Pyridine	110-86-1	8270 (3520)	0.81	50.00	7.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Safrole	94-59-7	8270 (3520)	0.99	10.00	NA	Not Available	NA
Sulfotepp	3689-24-5	8270 (3520)	0.70	10.00	NA	Not Available	NA
Thionazin	297-97-2	8270 (3520)	0.99	10.00	NA	Not Available	NA
Surrogates							
2-Fluorobiphenyl	321-60-8	8270 (3520)	NA	NA	NA	Not Available	NA
2-Fluorophenol	367-12-4	8270 (3520)	NA	NA	NA	Not Available	NA
Nitrobenzene-d5	98-95-3	8270 (3520)	NA	NA	3.50	IEPA TACO Class I	No
Phenol-d5	108-95-2	8270 (3520)	NA	NA	100.00	IEPA TACO Class I	No
Terphenyl-d14	26140-60-3	8270 (3520)	NA	NA	NA	Not Available	NA
2,4,6-Tribromophenol		8270 (3520)	NA	NA	NA	Not Available	NA
Non-Routine Analytes							
Atrazine	1912-24-9	8270 (3520)	0.89	10.00	3.00	IEPA TACO Class I	No

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Benzaldehyde	100-52-7	8270 (3520)	1.0	10.00	700.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
1,1-Biphenyl (1,1-Diphenyl)		8270 (3520)	0.68	10.00	NA	Not Available	NA
Caprolactam	105-60-2	8270 (3520)	0.84	10.00	3500.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
2,3-Dimethylphenol	526-75-0	8270 (3520)	0.86	10.00	NA	Not Available	NA
2,5-Dimethylphenol	95-87-4	8270 (3520)	0.93	10.00	NA	Not Available	NA
2,6-Dimethylphenol	576-26-1	8270 (3520)	0.69	10.00	NA	Not Available	NA
3,4-Dimethylphenol	95-65-8	8270 (3520)	0.79	10.00	NA	Not Available	NA
2,5-Dinitrophenol	329-71-5	8270 (3520)	0.99	50.00	NA	Not Available	NA
3-Nitrophenol	554-84-7	8270 (3520)	0.78	10.00	NA	Not Available	NA
Phenyl ether (Diphenyl oxide)	101-84-8	8270 (3520)	1.0	10.00	NA	Not Available	NA
1,2,3,5-Tetrachlorobenzene	634-90-2	8270 (3520)	0.75	10.00	NA	Not Available	NA
1,2,3-Trichlorobenzene	87-61-6	8270 (3520)	0.59	10.00	70.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
1,3,5-Trichlorobenzene	108-70-3	8270 (3520)	0.62	10.00	NA	Not Available	NA

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Soil to GW Screening Value (mg/L)	Ingestion Industrial-Commercial (mg/kg)	Inhalation Industrial-Commercial (mg/kg)	Ingestion Construction Worker (mg/kg)	Inhalation Construction Worker (mg/kg)	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Metals Parameters												
Aluminum	6010 (3050)	4.5	2.0	3.50	1,000,000	--	200,000	--	200,000.00	IEPA TACO - Class I	N.C.	No
Antimony	6010 (3050)	0.45	2.0	0.006	820	--	82	--	82.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Arsenic	6010 (3050)	0.67	1.0	0.05	--	1,200	61	25,000	61.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Barium	6010 (3050)	0.30	1.0	2.00	140,000	910,000	14,000	870,000	14,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Beryllium	6010 (3050)	0.017	0.40	0.004	4,100	21,000	410	44,000	410.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Boron	6010 (3050)	1.30	5.0	2.00	180,000	1,000,000	18,000	1,000,000	18,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Cadmium	6010 (3050)	0.22	0.50	0.005	2,000	2,800	200	59,000	200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Calcium	6010 (3050)	2.4	50	Not Available								
Chromium	6010 (3050)	0.13	1.0	0.10	6,100	420	4,100	690	420.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Cobalt	6010 (3050)	0.17	1.0	1.00	120,000	--	12,000	--	12,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Copper	6010 (3050)	0.17	2.0	0.65	82,000	--	8,200	--	8,200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Iron	6010 (3050)	4.2	5.0	5.00	--	--	--	--	0.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Lead	6010 (3050)	0.21	0.50	0.0075	400	--	400	--	400.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Magnesium	6010 (3050)	1.2	50	Not Available								
Manganese	6010 (3050)	0.21	1.0	0.15	96,000	91,000	9,600	8,700	8,700.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Molybdenum	6010 (3050)	0.33	1.0	Not Available								
Nickel	6010 (3050)	0.26	4.0	0.10	41,000	21,000	4,100	440,000	4,100.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Potassium	6010 (3050)	1.3	100	Not Available								
Selenium	6010 (3050)	0.90	2.5	0.05	10,000	--	1,000	--	1,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Silver	6010 (3050)	0.099	1.0	0.05	10,000	--	1,000	--	1,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Sodium	6010 (3050)	50	100	Not Available								
Strontium	6010 (3050)	0.15	1.0	4.20	1,000,000	--	120,000	--	120,000.00	IEPA TACO - Class I	N.C.	Yes

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Soil to GW Screening Value (mg/L)	Ingestion Industrial-Commercial (mg/kg)	Inhalation Industrial-Commercial (mg/kg)	Ingestion Construction Worker (mg/kg)	Inhalation Construction Worker (mg/kg)	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Thallium	6010 (3050)	1.30	2.5	0.002	160	--	160	--	160.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Tin	6010 (3050)	4.0	10	4.20	1,000,000	--	120,000	--	120,000.00	IEPA TACO - Class I	N.C.	No
Titanium	6010 (3050)	0.040	1.0	Not Available								
Vanadium	6010 (3050)	0.14	1.0	0.049	14,000	--	1,400	--	1,400.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Zinc	6010 (3050)	0.75	2.0	5.00	610,000	--	61,000	--	61,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Mercury	7471	0.0040	0.020	0.002	610	540,000	61	52,000	61.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
General Chemistry Parameters												
Ammonia (as N)	350.1(EPA-CE:3-140)	0.075	0.15	Not Available								
Polychlorinated Biphenyls as Aroclors by GC/EC (Sonication Extraction)												
PCB-1016	8082 (3550)	6.7	33	Not Available								
PCB 1221	8082 (3550)	6.8	67	Not Available								
PCB 1232	8082 (3550)	6.2	33	Not Available								
PCB-1242	8082 (3550)	7.5	33	Not Available								
PCB-1248	8082 (3550)	8.0	33	Not Available								
PCB-1254	8082 (3550)	5.2	33	Not Available								
PCB-1260	8082 (3550)	6.4	33	Not Available								
PCB-1268	8082 (3550)	11	33	Not Available								
Surrogates												
2,4,5,6-Tetrachloro-m-xylene	8082 (3550)	NA	NA	Not Available								
Decachlorobiphenyl	8082 (3550)	NA	NA	Not Available								
Chlorinated Pesticides by GC/EC (Sonication Extraction)												
4,4'-DDD	8081 (3550)	0.30	3.3	Not Available								
4,4'-DDE	8081 (3550)	0.30	3.3	Not Available								
4,4'-DDT (MS)	8081 (3550)	0.27	3.3	Not Available								
Aldrin (MS)	8081 (3550)	0.14	1.7	0.5	0.3	6.6	6.1	9.3	0.30	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
alpha Chlordane	8081 (3550)	0.17	1.7	Not Available								
alpha-BHC	8081 (3550)	0.52	1.7	Not Available								
beta-BHC	8081 (3550)	0.47	1.7	Not Available								
Chlorobenzilate	8081 (3550)	3.8	17	Not Available								
delta-BHC	8081 (3550)	0.23	1.7	Not Available								
Dieldrin (MS)	8081 (3550)	0.35	3.3	0.004	0.4	2.2	7.8	3.1	0.40	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Endosulfan I	8081 (3550)	0.16	1.7	18	12,000	--	1,200	--	1,200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Endosulfan II	8081 (3550)	0.27	3.3	18	12,000	--	1,200	--	1,200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Endosulfan sulfate	8081 (3550)	0.37	3.3	Not Available								
Endrin (MS)	8081 (3550)	0.32	3.3	1	610	--	61	--	61.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Endrin aldehyde	8081 (3550)	0.65	3.3	Not Available								
Endrin ketone	8081 (3550)	0.32	3.3	Not Available								
gamma Chlordane	8081 (3550)	0.22	1.7	Not Available								
gamma-BHC (Lindane) (MS)	8081 (3550)	0.14	1.7	Not Available								
Heptachlor (MS)	8081 (3550)	0.32	1.7	23	1	11	28	16	1.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Heptachlor epoxide	8081 (3550)	0.21	1.7	0.7	0.6	9.2	2.7	13	0.60	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Isodrin	8081 (3550)	0.33	3.3	Not Available								
Kepone	8081 (3550)	6.7	170	Not Available								
Methoxychlor	8081 (3550)	0.47	17	160	10,000	--	1,000	--	1,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Technical Chlordane	8081 (3550)	3	17	Not Available								
Toxaphene	8081 (3550)	12	170	31	5.2	170	110	240	5.20	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Surrogates												
Tetrachloro-m-xylene	8081 (3550)	NA	NA	Not Available								
Decachlorobiphenyl	8081 (3550)	NA	NA	Not Available								
Non-routine Compounds												
2,4'-DDD	8081 (3550)	3.3	3.3	Not Available								
2,4'-DDE	8081 (3550)	3.3	3.3	Not Available								
2,4'-DDT	8081 (3550)	3.3	3.3	Not Available								
Chlorinated Herbicides by GC/EC												
2,4-D (MS)	8151	0.97	8.3	1.5	20,000	--	2,000	--	2,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,4-DB	8151	1.0	8.3	Not Available								
2,4,5-T (MS)	8151	0.56	8.3	Not Available								
2,4,5-TP (Silvex) (MS)	8151	0.52	8.3	11	16,000	--	1,600	--	1,600.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Dalapon	8151	10	330	0.85	61,000	--	6,100	--	6,100.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Dicamba	8151	1.5	8.3	2.8	61,000	--	6,100	--	6,100.00	IEPA TACO - Class I	No	No
Dichlorprop	8151	1.8	8.3	Not Available								

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Dinoseb	8151	14	100	0.34	2,000	--	200	--	200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
MCPA	8151	42	2000		1,000	--	100	--	100.00	IEPA TACO - Class I	No	No
MCPP	8151	220	2000		2,000	--	2,000	--	2,000.00	IEPA TACO - Class I	No	No
Pentachlorophenol	8151	0.98	8.3	0.03	24	--	520	--	24.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Surrogates												
2,4-Dichlorophenyl acetic acid	8151	NA	NA	Not Available								
Non-Routine Compounds												
Picloram	8151	2.6	8.3	2	140,000	--	14,000	--	14,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Volatiles by GC/MS												
1,1,1,2-Tetrachloroethane	8260 (5035)	0.45	5.0	2	61,000	1,000	6,100	1,000	1,000.00	IEPA TACO - Class I	No	No
1,1,1-Trichloroethane	8260 (5035)	0.50	5.0	2	--	1,200	--	1,200	1,200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
1,1,2,2-Tetrachloroethane	8260 (5035)	0.60	5.0	3.3	120,000	2,000	12,000	2,000	2,000.00	IEPA TACO - Class I	No	No
1,1,2-Trichloro-1,2,2-trifluoroethane	8260 (5035)	1.2	5.0	Not Available								
1,1,2-Trichloroethane	8260 (5035)	0.54	5.0	0.02	8,200	1,800	8,200	1,800	1,800.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
1,1-Dichloroethane	8260 (5035)	0.4	5.0	23	200,000	1,700	200,000	130	130.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
1,1-Dichloroethene (MS)	8260 (5035)	0.56	5.0	Not Available								
1,1-Dichloropropene	8260 (5035)	0.64	5.0	Not Available								
1,2,3-Trichlorobenzene	8260 (5035)	0.38	5.0	5.7	20,000	--	2,000	--	2,000.00	IEPA TACO - Class I	Yes	No
1,2,3-Trichloropropane	8260 (5035)	0.87	5.0	0.0001	0.82	1,000	18	1,000	0.82	IEPA TACO - Class I	No	No
1,2,4-Trichlorobenzene	8260 (5035)	0.46	5.0	5	20,000	3,200	2,000	920	920.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
1,2,4-Trimethylbenzene	8260 (5035)	0.4	5.0	18	100,000	110	10,000	0.73	0.73	IEPA TACO - Class I	Yes	No
1,2-Dibromo-3-chloropropane (DBCP)	8260 (5035)	1.0	10	0.002	4	17	89	0.11	0.11	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
1,2-Dibromoethane (EDB)	8260 (5035)	0.73	5.0	0.0004	0.07	0.32	1.5	0.45	0.07	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
1,2-Dichlorobenzene	8260 (5035)	0.53	5.0	17	180,000	560	18,000	310	310.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
1,2-Dichloroethane	8260 (5035)	0.59	5.0	0.02	63	0.7	1,400	0.99	0.70	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
1,2-Dichloroethenes (total)	8260 (5035)	0.62	10	Not Available								
1,2-Dichloropropane	8260 (5035)	0.42	5.0	0.03	84	23	1,800	0.5	0.50	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
1,3,5-Trimethylbenzene	8260 (5035)	0.34	5.0	10	100,000	71	10,000	0.46	0.46	IEPA TACO - Class I	Yes	No
1,3-Dichlorobenzene	8260 (5035)	0.5	5.0	0.2	1,800	570	180	570	180.00	IEPA TACO - Class I	No	No

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
1,3-Dichloropropane	8260 (5035)	0.56	5.0	Not Available								
1,4-Dichlorobenzene	8260 (5035)	0.47	5.0	2	--	17,000	--	340	340.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,2-Dichloropropane	8260 (5035)	0.59	5.0	Not Available								
2-Butanone (Methyl Ethyl Ketone-MEK)	8260 (5035)	1.8	25	17	1,000,000	25,000	120,000	710	710.00	IEPA TACO - Class I	No	No
2-Chloro-1,3-butadiene (Chloroprene)	8260 (5035)	2.1	5.0	Not Available								
2-Chloroethyl vinyl ether	8260 (5035)	50.0	50	Not Available								
2-Chlorotoluene	8260 (5035)	0.5	5.0	Not Available								
2-Hexanone	8260 (5035)	2.0	25	1.3	82,000	110	8,200	0.72	0.72	IEPA TACO - Class I	No	No
3-Chloropropene (Allyl chloride)	8260 (5035)	2.3	5.0	Not Available								
4-Chlorotoluene	8260 (5035)	0.57	5.0	Not Available								
4-Methyl-2-pentanone (MIBK)	8260 (5035)	1.9	25	Not Available								
Acetone	8260 (5035)	2.6	50	16	200,000	100,000	200,000	100,000	100,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Acetonitrile	8260 (5035)	53	200	Not Available								
Acrolein	8260 (5035)	23	100	0.014	1,000	0.26	100	0.0017	0.0017	IEPA TACO - Class I	No	No
Acrylonitrile	8260 (5035)	16	100	0.0006	11	0.54	230	0.17	0.17	IEPA TACO - Class I	No	No
Benzene (MS)	8260 (5035)	0.52	5.0	0.03	100	1.6	2,300	2.2	1.60	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Bromobenzene	8260 (5035)	0.4	5.0	Not Available								
Bromochloromethane	8260 (5035)	0.47	5.0	Not Available								
Bromodichloromethane	8260 (5035)	0.42	5.0	0.6	92	3,000	2,000	3,000	92.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Bromoform	8260 (5035)	0.62	5.0	0.8	720	100	16,000	140	100.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Bromomethane	8260 (5035)	4.3	5.0	0.2	2,900	15	1,000	3.9	3.90	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Carbon disulfide	8260 (5035)	0.4	5.0	32	200,000	720	20,000	9	9.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Carbon tetrachloride	8260 (5035)	0.36	5.0	0.07	44	0.64	410	0.9	0.64	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Chlorobenzene (MS)	8260 (5035)	0.84	5.0	1	41,000	210	4,100	1.3	1.30	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Chloroethane	8260 (5035)	1.0	5.0	15	820,000	1,500	82,000	94	94.00	IEPA TACO - Class I	Yes	No
Chloroform	8260 (5035)	0.36	5.0	0.6	940	0.54	2,000	0.76	0.54	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Chloromethane	8260 (5035)	0.42	5.0	0.14	8,200	170	820	1.1	1.10	IEPA TACO - Class I	No	No
cis-1,2-Dichloroethene	8260 (5035)	0.30	5.0	Not Available								
cis-1,3-Dichloropropene	8260 (5035)	0.45	5.0	0.004	57	2.1	1,200	0.39	0.39	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Dibromochloromethane	8260 (5035)	0.5	5.0	0.4	41,000	1,300	41,000	1,300	1,300.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Dibromomethane	8260 (5035)	0.42	5.0	Not Available								
Dichlorodifluoromethane	8260 (5035)	0.90	5.0	48	410,000	390	180,000	26	26.00	IEPA TACO - Class I	Yes	No
Ethyl methacrylate	8260 (5035)	1.2	5.0	Not Available								
Ethylbenzene	8260 (5035)	0.6	5.0	13	200,000	400	20,000	58	58.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Hexachlorobutadiene	8260 (5035)	0.68	5.0	Not Available								
Iodomethane	8260 (5035)	1.3	5.0	Not Available								
Isobutyl alcohol	8260 (5035)	83	200	Not Available								
Isopropylbenzene	8260 (5035)	0.35	5.0	86	200,000	760	20,000	4.9	4.90	IEPA TACO - Class I	Yes	No
m&p-Xylene	8260 (5035)	1.5	5.0	200	1,000,000	420	410,000	420	420.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Methacrylonitrile	8260 (5035)	31	100	Not Available								
Methyl methacrylate	8260 (5035)	2.0	5.0	Not Available								
Methyl t-butyl ether (MTBE)	8260 (5035)	0.69	50	Not Available								
Methylene chloride	8260 (5035)	0.87	5.0	0.02	760	24	12,000	34	24.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Naphthalene	8260 (5035)	1.8	5.0	12	41,000	270	4,100	1.8	1.80	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
n-Butylbenzene	8260 (5035)	0.42	5.0	11	20,000	53	2,000	53	53.00	IEPA TACO - Class I	Yes	No
n-Propylbenzene	8260 (5035)	0.47	5.0	2.6	20,000	260	2,000	260	260.00	IEPA TACO - Class I	No	No
o-Xylene	8260 (5035)	0.44	5.0	190	1,000,000	410	410,000	410	410.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Pentachloroethane	8260 (5035)	1.4	25	Not Available								
p-Isopropyltoluene	8260 (5035)	0.39	5.0	Not Available								
Propionitrile (ethylcyanide)	8260 (5035)	30	100	Not Available								
sec-Butylbenzene	8260 (5035)	0.45	5.0	15	20,000	42	2,000	42	42.00	IEPA TACO - Class I	Yes	No
Styrene	8260 (5035)	0.53	5.0	4	410,000	1,500	41,000	430	430.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
tert-Butylbenzene	8260 (5035)	0.52	5.0	6.5	20,000	77	2,000	77	77.00	IEPA TACO - Class I	Yes	No
Tetrachloroethene	8260 (5035)	0.91	5.0	Not Available								
Toluene (MS)	8260 (5035)	0.69	5.0	12	410,000	650	410,000	42	42.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
trans-1,2-Dichloroethene	8260 (5035)	0.37	5.0	Not Available								
trans-1,3-Dichloropropene	8260 (5035)	0.45	5.0	0.004	57	2.1	1,200	0.39	0.39	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
trans-1,4-Dichloro-2-butene	8260 (5035)	3.6	10	Not Available								
Trichloroethene (MS)	8260 (5035)	4.4	5.0	Not Available								
Trichlorofluoromethane	8260 (5035)	0.4	5.0	33	610,000	1,300	410,000	85	85.00	IEPA TACO - Class I	Yes	No
Vinyl acetate	8260 (5035)	0.83	10	170	1,000,000	1,600	200,000	10	10.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Vinyl chloride	8260 (5035)	0.59	5.0	0.01	7.9	1.1	170	1.1	1.10	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Xylenes (total)	8260 (5035)	1.9	10	150	1,000,000	320	410,000	320	320.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Surrogates												
p-Bromofluorobenzene	8260 (5035)	NA	NA	Not Available								
Dibromofluoromethane	8260 (5035)	NA	NA	Not Available								
Toluene-d8	8260 (5035)	NA	NA	Not Available								
Non-Routine Compounds												
1-Chlorohexane	8260 (5035)	0.72	5.0	Not Available								
Cyclohexane	8260 (5035)	10	10	Not Available								
Cyclohexanone	8260 (5035)	15	50	150	1,000,000	660	1,000,000	660	660.00	IEPA TACO - Class I	Yes	No
Diethyl ether	8260 (5035)	0.78	10	Not Available								
Furan	8260 (5035)	0.36	5.0	Not Available								
Methyl acetate	8260 (5035)	1.6	10	7	1,000,000	27,000	1,000,000	27,000	27,000.00	IEPA TACO - Class I	No	No
Methyl cyclohexane	8260 (5035)	0.48	10	Not Available								
Tetrahydrofuran	8260 (5035)	0.36	10	Not Available								
Semivolatiles (Base-Neutrals/Acids) by GC/MS (Sonication Extraction)												
1,2,4,5-Tetrachlorobenzene	8270 (3550)	31	330	Not Available								
1,2,4-Trichlorobenzene (MS)	8270 (3550)	21	330	Not Available								
1,2-Dichlorobenzene	8270 (3550)	23	330	Not Available								
1,2-Diphenyl hydrazine	8270 (3550)	22	330	Not Available								
1,3,5-Trinitrobenzene	8270 (3550)	82	330	0.97	61,000	--	6,100	--	6,100.00	IEPA TACO - Class I	No	No
1,3-Dichlorobenzene	8270 (3550)	26	330	Not Available								
1,4-Dichlorobenzene (MS)	8270 (3550)	22	330	Not Available								
1,4-Dioxane	8270 (3550)	66	330	0.031	520	100,000	11,000	98,000	520.00	IEPA TACO - Class I	No	No
1,4-naphthoquinone	8270 (3550)	39	330	Not Available								
1-Methylnaphthalene	8270 (3550)	29	330	7.2	8,200	990	820	990	820.00	IEPA TACO - Class I	No	No
1-Naphthylamine	8270 (3550)	82	330	Not Available								
2,4,5-Trichlorophenol	8270 (3550)	32	330	270	200,000	--	200,000	--	200,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,4,6-Trichlorophenol	8270 (3550)	20	330	0.2	520	390	11,000	540	390.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,4-Dichlorophenol	8270 (3550)	23	330	1	6,100	--	610	--	610.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,4-Dimethylphenol	8270 (3550)	35	330	9	41,000	--	41,000	--	41,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,4-Dinitrophenol	8270 (3550)	170	1700	0.2	4,100	--	410	--	410.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,4-Dinitrotoluene (MS)	8270 (3550)	19	330	0.0008	8.4	--	180	--	8.40	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,6-Dichlorophenol	8270 (3550)	53	330	Not Available								

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2,6-Dinitrotoluene	8270 (3550)	19	330	0.0007	8.4	--	180	--	8.40	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2-Acetylaminofluorene	8270 (3550)	38	330	Not Available								
2-Chloronaphthalene	8270 (3550)	24	330	Not Available								
2-Chlorophenol (MS)	8270 (3550)	27	330	4	10,000	53,000	10,000	53,000	10,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2-Methyl phenol (o-Cresol)	8270 (3550)	30	330	15	100,000	--			100,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2-Methylnaphthalene	8270 (3550)	24	330	7.7	8,200	--	820	--	820.00	IEPA TACO - Class I	No	No
2-Naphthylamine	8270 (3550)	82	330	Not Available								
2-Nitroaniline	8270 (3550)	23	1700	--	--	120	--	7.5	7.50	IEPA TACO - Class I	Yes	No
2-Nitrophenol	8270 (3550)	20	330	Not Available								
2-Picoline	8270 (3550)	82	330	Not Available								
3- and 4-Methyl phenol	8270 (3550)	29	330	Not Available								
3,3'-Dichlorobenzidine	8270 (3550)	420	660	0.007	13	--			13.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
3,3'-Dimethylbenzidine	8270 (3550)	30	1700	Not Available								
3-Methylcholanthrene	8270 (3550)	26	330	Not Available								
3-Nitroaniline	8270 (3550)	33	1700	Not Available								
4,6-Dinitro-2-methylphenol	8270 (3550)	200	1700	Not Available								
4-Aminobiphenyl	8270 (3550)	54	330	Not Available								
4-Bromophenyl phenyl ether	8270 (3550)	31	330	Not Available								
4-Chloro-3-methylphenol (MS)	8270 (3550)	34	330	Not Available								
4-Chloroaniline	8270 (3550)	26	660	0.7	8,200	--	820	--	820.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
4-Chlorophenylphenyl ether	8270 (3550)	19	330	Not Available								
4-Nitroaniline	8270 (3550)	17	1700	Not Available								
4-Nitrophenol (MS)	8270 (3550)	210	1700	Not Available								
4-Nitroquinoline-1-oxide	8270 (3550)	166	3300	Not Available								
7,12-Dimethylbenz(a)anthracene	8270 (3550)	31	330	Not Available								
a,a-Dimethylphenethylamine	8270 (3550)	490	6700	Not Available								
Acenaphthene (MS)	8270 (3550)	19	330	570	120,000	--	120,000	--	120,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Acenaphthylene	8270 (3550)	17	330	24	61,000	--			61,000.00	IEPA TACO - Class I	No	No
Acetophenone	8270 (3550)	22	330	Not Available								
Aniline	8270 (3550)	21	330	0.064	1,000	130	8.6	0.064	0.06	IEPA TACO - Class I	No	No
Anthracene	8270 (3550)	23	330	12,000	610,000	--	610,000	--	610,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Aramite	8270 (3550)	60	330	Not Available								
Benzidine	8270 (3550)	83	2700	4.30E-06	0.02	0.02	0.54	0.02	0.02	IEPA TACO - Class I	No	No
Benzo(a)anthracene	8270 (3550)	31	330	2	8	--	170	--	8.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No

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Benzo(a)pyrene	8270 (3550)	19	330	8	0.8	--	17	--	0.80	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Benzo(b)fluoranthene	8270 (3550)	26	330	5	8	--	170	--	8.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Benzo(g,h,i)perylene	8270 (3550)	23	330	32,000	61,000	--	61,000	--	61,000.00	IEPA TACO - Class I	Yes	No
Benzo(k)fluoranthene	8270 (3550)	36	330	49	78	--	1,700	--	78.00		No	No
Benzoic acid	8270 (3550)	170	1700	400	1,000,000	--	820,000	--	820,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Benzyl alcohol	8270 (3550)	38	330	9.4	610,000	6,900	200,000	6,900	6,900.00	IEPA TACO - Class I	No	No
Bis(2-chloroethoxy) methane	8270 (3550)	24	330	Not Available								
Bis(2-chloroethyl) ether	8270 (3550)	28	330	0.0004	5	0.47	75	1	0.47	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Bis(2-chloroisopropyl) ether	8270 (3550)	35	330	2.4	82,000	1,300			1,300.00	IEPA TACO - Class I	No	No
Bis(2-ethylhexyl) phthalate	8270 (3550)	38	330	3,600	410	31,000	4,100	31,000	410.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Butyl benzyl phthalate	8270 (3550)	27	330	930	410,000	930	410,000	930	930.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Carbazole	8270 (3550)	28	330	0.6	290	--	6,200	--	290.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Chrysene	8270 (3550)	25	330	160	780	--	17,000	--	780.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Diallate	8270 (3550)	56	330	Not Available								
Dibenzo(a,h)anthracene	8270 (3550)	24	330	2	0.8	--	17	--	0.80	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Dibenzofuran	8270 (3550)	19	330	15	8,200	--	820	--	820.00	IEPA TACO - Class I	No	No
Diethylphthalate	8270 (3550)	22	330	470	1,000,000	2,000	1,000,000	2,000	2,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Dimethoate	8270 (3550)	45	330								No	No
Dimethylphthalate	8270 (3550)	19	330	380	1,000,000	1,300	1,000,000	1,300	1,300.00	IEPA TACO - Class I	Yes	No
Di-n-butylphthalate	8270 (3550)	28	330	2,300	200,000	2,300	200,000	2,300	2,300.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Di-n-octylphthalate	8270 (3550)	31	330	10,000	41,000	10,000	4,100	10,000	4,100.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	8270 (3550)	83	330	Not Available								
Diphenylamine/ N-nitrosodiphenylamine	8270 (3550)	23	330	20	51,000	--	5,100	--	5,100.00	IEPA TACO - Class I	No	No
Disulfoton	8270 (3550)	55	330	0.097	82	820	8.2	820	8.20	IEPA TACO - Class I	No	No
Ethyl methanesulfonate	8270 (3550)	66	330	Not Available								
Ethyl parathion	8270 (3550)	55	330	Not Available								
Famphur	8270 (3550)	98	330	Not Available								
Fluoranthene	8270 (3550)	26	330	4,300	82,000	--	82,000	--	82,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No

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Fluorene	8270 (3550)	22	330	560	82,000	--	82,000	--	82,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Hexachlorobenzene	8270 (3550)	26	330	2	4	1.8	78	2.6	1.80	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Hexachlorobutadiene	8270 (3550)	20	330	2.9	410	1,000	41	180	41.00	IEPA TACO - Class I	No	No
Hexachlorocyclopentadiene	8270 (3550)	83	330	400	14,000	16	14,000	1.1	1.10	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Hexachloroethane	8270 (3550)	20	330	0.5	2,000	--	2,000	--	2,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Hexachlorophene	8270 (3550)	12500	170000	Not Available								
Hexachloropropene	8270 (3550)	45	330	Not Available								
Indeno(1,2,3-cd)pyrene	8270 (3550)	26	330	14	8	--	170	--	8.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Isophorone	8270 (3550)	22	330	8	410,000	4,600	410,000	4,600	4,600.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Isosafrole	8270 (3550)	64	330	Not Available								
m-Dinitrobenzene	8270 (3550)	50	330	Not Available								
Methapyrilene	8270 (3550)	42	67000	Not Available								
Methyl methanesulfonate	8270 (3550)	55	330	Not Available								
Methyl parathion	8270 (3550)	47	330	Not Available								
Naphthalene	8270 (3550)	19	330	12	41,000	270			270.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Nitrobenzene	8270 (3550)	35	330	0.1	1,000	140	1,000	9.4	9.40	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
N-Nitro-o-toluidine	8270 (3550)	32	330	Not Available								
N-Nitrosodiethylamine	8270 (3550)	44	330	Not Available								
N-Nitrosodimethylamine	8270 (3550)	59	330	Not Available								
N-Nitroso-di-N-butylamine	8270 (3550)	52	330	Not Available								
N-Nitrosodiphenylamine/ Diphenylamine	8270 (3550)	23	330	1	1,200	--	25,000	--	1,200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
N-Nitrosomethylethylamine	8270 (3550)	52	330	Not Available								
N-Nitrosomorpholine	8270 (3550)	66	330	Not Available								
N-Nitrosopiperidine	8270 (3550)	71	330	Not Available								
N-Nitrosopyrrolidine	8270 (3550)	45	330	Not Available								
N-Nitrosos-di-N-propylamine (MS)	8270 (3550)	28	330								No	No
o,o,o-Triethylphosphorothioate	8270 (3550)	72	330	Not Available								
o-Toluidine	8270 (3550)	56	330	Not Available								
p-(Dimethylamino)azobenzene	8270 (3550)	38	330	Not Available								
Pentachlorobenzene	8270 (3550)	64	330	Not Available								
Pentachloronitrobenzene	8270 (3550)	56	330	28	6,100	--	610	--	610.00	IEPA TACO - Class I	No	No
Pentachlorophenol (MS)	8270 (3550)	83	1700	Not Available								
Phenacetin	8270 (3550)	51	330	Not Available								
Phenanthrene	8270 (3550)	29	330	220	61,000	--	61,000	--	61,000.00	IEPA TACO - Class I	No	No

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Phenol (MS)	8270 (3550)	30	330	100	1,000,000	--	120,000	--	120,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Phorate	8270 (3550)	69	330	0.31	410	1,600	41	710	41.00	IEPA TACO - Class I	No	No
p-Phenylenediamine	8270 (3550)	350	1700	Not Available								
Pronamide	8270 (3550)	63	330	Not Available								
Pyrene (MS)	8270 (3550)	20	330	4,200	61,000	--	61,000	--	61,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Pyridine	8270 (3550)	30	330	0.029	2,000	120,000	2,000	5,200	2,000.00	IEPA TACO - Class I	No	No
Safrole	8270 (3550)	53	330	Not Available								
Sulfotepp	8270 (3550)	55	330	Not Available								
Tetrachlorophenols (2,3,4,5 + 2,3,4,6)	8270 (3550)	45	330	Not Available								
Thionazin	8270 (3550)	42	330	Not Available								
Surrogates												
2-Fluorobiphenyl	8270 (3550)	NA	NA	Not Available								
2-Fluorophenol	8270 (3550)	NA	NA	Not Available								
Nitrobenzene-d5	8270 (3550)	NA	NA	Not Available								
Phenol-d5	8270 (3550)	NA	NA	Not Available								
p-Terphenyl-d14	8270 (3550)	NA	NA	Not Available								
2,4,6- Tribromophenol	8270 (3550)	NA	NA	Not Available								

Notes:

1) Shadded chemicals indicate the chemical is not included in the Superfund Target Compound or Analyte List.

2) N.C. = Not Comparable.

Table 4.3
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (ug/L)	RL (ug/L)
Metals						
Aluminum	6010 (3010)	1	75-125	<=20	30	200
Antimony	6010 (3010)	1	75-125	<=20	5.1	20
Arsenic	6010 (3010)	1	75-125	<=20	5.2	10
Barium	6010 (3010)	1	75-125	<=20	1.2	10
Beryllium	6010 (3010)	1	75-125	<=20	0.14	4.0
Cadmium	6010 (3010)	1	75-125	<=20	1.0	5.0
Calcium	6010 (3010)	1	75-125	<=20	50	500
Chromium	6010 (3010)	1	75-125	<=20	0.95	10
Cobalt	6010 (3010)	1	75-125	<=20	1.3	10
Copper	6010 (3010)	1	75-125	<=20	1.7	20
Iron	6010 (3010)	1	75-125	<=20	28	50
Lead	6010 (3010)	1	75-125	<=20	3.5	5.0
Magnesium	6010 (3010)	1	75-125	<=20	9.8	500
Manganese	6010 (3010)	1	75-125	<=20	2.0	10
Nickel	6010 (3010)	1	75-125	<=20	1.5	40
Potassium	6010 (3010)	1	75-125	<=20	15	1000
Selenium	6010 (3010)	1	75-125	<=20	5.4	10
Silver	6010 (3010)	1	75-125	<=20	1.0	10
Sodium	6010 (3010)	1	75-125	<=20	300	1000
Thallium	6010 (3010)	1	75-125	<=20	11	25
Tin	6010 (3010)	1	75-125	<=20	6.3	50
Vanadium	6010 (3010)	1	75-125	<=20	1.4	10
Zinc	6010 (3010)	1	75-125	<=20	3.0	20
Mercury (CVAA)	7470	1	80-120	<=20	0.080	0.20
Polychlorinated Biphenyls as Aroclors by GC/EC						
PCB 1016 (MS)	8082 (3520)	1	43-114	<=40	0.14	1.0
PCB 1221	8082 (3520)	1	30-110	<=40	0.50	2.0
PCB 1232	8082 (3520)	1	30-110	<=40	0.22	1.0
PCB 1242	8082 (3520)	1	30-110	<=40	0.22	1.0
PCB 1248	8082 (3520)	1	30-110	<=40	0.33	1.0
PCB 1254	8082 (3520)	1	30-110	<=40	0.20	1.0
PCB 1260 (MS)	8082 (3520)	1	52-121	<=40	0.17	1.0
PCB 1268	8082 (3520)	1	40-130	<=40	0.10	1.0
Surrogates						
Decachlorobiphenyl	8082 (3520)	1	30-150	NA	NA	NA
2,4,5,6-Tetrachloro-m-xylene	8082 (3520)	1	30-150	NA	NA	NA
Chlorinated Pesticides in Groundwater by GC/EC						
4,4'-DDD	8081 (3520)	1	59-141	<=40	0.011	0.10
4,4'-DDE	8081 (3520)	1	44-117	<=40	0.012	0.10
4,4'-DDT (MS)	8081 (3520)	1	39-140	<=40	0.014	0.10
Aldrin (MS)	8081 (3520)	1	37-120	<=40	0.0053	0.050
alpha BHC	8081 (3520)	1	31-112	<=40	0.0086	0.050
alpha Chlordane	8081 (3520)	1	45-120	<=40	0.0068	0.050
beta BHC	8081 (3520)	1	19-131	<=40	0.0057	0.050
Chlorobenzilate	8081 (3520)	1	35-155	<=40	0.19	0.50

Table 4.3
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (ug/L)	RL (ug/L)
Chlorinated Pesticides in Groundwater by GC/EC						
delta BHC	8081 (3520)	1	39-123	<=40	0.0098	0.050
Dieldrin (MS)	8081 (3520)	1	38-136	<=40	0.010	0.10
Endosulfan I	8081 (3520)	1	40-119	<=40	0.0064	0.050
Endosulfan II	8081 (3520)	1	40-123	<=40	0.011	0.10
Endosulfan sulfate	8081 (3520)	1	54-132	<=40	0.010	0.10
Endrin (MS)	8081 (3520)	1	34-146	<=40	0.010	0.10
Endrin aldehyde	8081 (3520)	1	33-142	<=40	0.017	0.10
Endrin ketone	8081 (3520)	1	37-165	<=40	0.016	0.10
gamma BHC (Lindane) (MS)	8081 (3520)	1	27-115	<=40	0.0055	0.050
gamma Chlordane	8081 (3520)	1	55-123	<=40	0.013	0.050
Heptachlor (MS)	8081 (3520)	1	35-121	<=40	0.0076	0.050
Heptachlor epoxide	8081 (3520)	1	49-120	<=40	0.015	0.050
Isodrin	8081 (3520)	1	24-214	<=40	0.0082	0.050
Kepone	8081 (3520)	1	D-105	<=100	0.12	1.0
Methoxychlor	8081 (3520)	1	53-167	<=40	0.024	0.50
Toxaphene	8081 (3520)	1	39-137	<=40	0.88	5.0
Surrogates						
Non-routine Compounds						
2,4'-DDD	8081 (3520)	1	59-141	<=40	0.073	0.10
2,4'-DDE	8081 (3520)	1	44-117	<=40	0.068	0.10
2,4'-DDT	8081 (3520)	1	39-140	<=40	0.071	0.10
Chlorinated Herbicides by GC/EC						
2,4,5-T (MS)	8151	1	38-138	<=40	0.080	0.50
2,4,5-TP (Silvex) (MS)	8151	1	44-118	<=40	0.033	0.50
2,4-D (MS)	8151	1	45-146	<=40	0.11	0.50
2,4-DB	8151	1	35-140	<=40	0.057	0.50
Dalapon	8151	1	48-187	<=40	0.24	10
Dicamba	8151	1	60-113	<=40	0.048	0.50
Dichlorprop	8151	1	43-106	<=40	0.030	0.50
Dinoseb	8151	1	23-117	<=40	0.40	6.0
MCPA	8151	1	47-131	<=40	12	120
MCPP	8151	1	27-150	<=40	10	120
Pentachlorophenol	8151	1	46-144	<=40	0.18	0.25
Volatiles in Groundwater by GC/MS						
1,1,1,2-Tetrachloroethane	8260 (5030)	1	62-107	<=30	0.53	1.0
1,1,1-Trichloroethane	8260 (5030)	1	70-132	<=30	0.79	1.0
1,1,2,2-Tetrachloroethane	8260 (5030)	1	71-127	<=30	0.21	1.0
1,1,2-Trichloroethane	8260 (5030)	1	75-122	<=30	0.37	1.0
1,1-Dichloroethane	8260 (5030)	1	70-127	<=30	0.56	1.0
1,1-Dichloroethene (MS)	8260 (5030)	1	64-132	<=30	0.93	1.0
1,2,3-Trichloropropane	8260 (5030)	1	60-147	<=30	0.44	1.0
1,2,4-Trichlorobenzene	8260 (5030)	1	48-131	<=30	0.28	1.0
1,2-Dibromo-3-chloropropane	8260 (5030)	1	14-147	<=50	0.65	1.0
1,2-Dibromoethane	8260 (5030)	1	60-118	<=30	0.34	1.0
1,2-Dichlorobenzene	8260 (5030)	1	71-125	<=30	0.21	1.0
1,2-Dichloroethane	8260 (5030)	1	68-130	<=30	0.28	1.0

Table 4.3
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Volatiles in Groundwater by GC/MS						
1,2-Dichloroethenes, Total (sum of cis- and trans- isomers)	8260 (5030)	1	70-130	<=30	1.2	2.0
1,2-Dichloropropane	8260 (5030)	1	74-123	<=30	0.26	1.0
1,3-Dichlorobenzene	8260 (5030)	1	70-125	<=30	0.28	1.0
1,4-Dichlorobenzene	8260 (5030)	1	65-127	<=30	0.44	1.0
2-Butanone (methyl ethyl ketone-MEK)	8260 (5030)	1	51-142	<=30	0.72	10
2-Chloro-1,3-butadiene (Chloroprene)	8260 (5030)	1	70-130	<=30	0.40	1.0
2-Hexanone	8260 (5030)	1	58-139	<=30	0.39	10
3-Chloropropene (Allyl chloride)	8260 (5030)	1	D-200	<=100	0.35	1.0
4-Methyl-2-pentanone (MIBK)	8260 (5030)	1	62-130	<=30	0.45	10
Acetone	8260 (5030)	1	20-183	<=50	7.3	25
Acetonitrile	8260 (5030)	1	71-158	<=30	0.23	40
Acrolein	8260 (5030)	1	40-91	<=30	12	20
Acrylonitrile	8260 (5030)	1	46-144	<=30	3.2	20
Benzene (MS)	8260 (5030)	1	74-122	<=30	0.54	1.0
Bromodichloromethane	8260 (5030)	1	74-128	<=30	0.42	1.0
Bromoform	8260 (5030)	1	64-132	<=30	0.36	1.0
Bromomethane	8260 (5030)	1	21-176	<=50	0.93	1.0
Carbon disulfide	8260 (5030)	1	60-130	<=30	0.75	1.0
Carbon tetrachloride	8260 (5030)	1	64-137	<=30	0.91	1.0
Chlorobenzene (MS)	8260 (5030)	1	75-123	<=30	0.41	1.0
Chloroethane	8260 (5030)	1	40-171	<=50	0.89	1.0
Chloroform	8260 (5030)	1	74-124	<=30	0.52	1.0
Chloromethane	8260 (5030)	1	51-133	<=50	0.53	1.0
cis-1,2-Dichloroethene	8260 (5030)	1	69-126	<=30	0.55	1.0
cis-1,3-Dichloropropene	8260 (5030)	1	76-126	<=30	0.25	1.0
Dibromochloromethane	8260 (5030)	1	75-126	<=30	0.40	1.0
Dibromomethane	8260 (5030)	1	70-130	<=30	0.33	1.0
Dichlorodifluoromethane	8260 (5030)	1	70-130	<=50	0.73	1.0
Ethyl methacrylate	8260 (5030)	1	58-101	<=30	0.33	1.0
Ethylbenzene	8260 (5030)	1	77-123	<=30	0.62	1.0
Hexachlorobutadiene	8260 (5030)	1	58-133	<=30	0.50	1.0
Iodomethane	8260 (5030)	1	34-116	<=30	0.96	5.0
Isobutyl alcohol	8260 (5030)	1	39-132	<=30	13	40
m&p-Xylene	8260 (5030)	1	74-123	<=30	1.3	2.0
Methacrylonitrile	8260 (5030)	1	65-110	<=30	9.1	20
Methylene chloride	8260 (5030)	1	67-128	<=30	0.44	5.0
Methylmethacrylate	8260 (5030)	1	57-120	<=30	0.36	1.0
Naphthalene	8260 (5030)	1	58-143	<=30	0.12	5.0
o-Xylene	8260 (5030)	1	76-122	<=30	0.49	1.0
Pentachloroethane	8260 (5030)	1	1-200	<=100	1.0	5.0
Propionitrile (ethylcyanide)	8260 (5030)	1	72-121	<=30	10	20
Styrene	8260 (5030)	1	75-125	<=30	0.42	1.0
Tetrachloroethene	8260 (5030)	1	70-133	<=30	0.75	1.0
Toluene (MS)	8260 (5030)	1	75-122	<=30	0.62	1.0
trans-1,2-Dichloroethene	8260 (5030)	1	67-130	<=30	0.80	1.0
trans-1,3-Dichloropropene	8260 (5030)	1	75-126	<=30	0.36	1.0

Table 4.3
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Volatiles in Groundwater by GC/MS						
trans-1,4-Dichloro-2-butene	8260 (5030)	1	26-131	<=50	0.80	2.0
Trichloroethene(MS)	8260 (5030)	1	75-122	<=30	0.71	1.0
Trichlorofluoromethane	8260 (5030)	1	74-165	<=50	0.96	1.0
Vinyl acetate	8260 (5030)	1	47-150	<=30	0.70	2.0
Vinyl chloride	8260 (5030)	1	59-136	<=50	0.92	1.0
Xylenes (total)	8260 (5030)	1	77-121	<=30	1.6	2.0
Semivolatiles in Groundwater by GC/MS						
1,2,4,5-Tetrachlorobenzene	8270 (3520)	1	17-127	<=40	1.2	10
1,2,4-Trichlorobenzene (MS)	8270 (3520)	1	46-99	<=40	0.54	10
1,2-Dichlorobenzene	8270 (3520)	1	42-98	<=40	1.0	10
1,3,5-Trinitrobenzene	8270 (3520)	1	10-123	<=40	0.62	10
1,3-Dichlorobenzene	8270 (3520)	1	38-97	<=40	0.55	10
1,4-Dichlorobenzene(MS)	8270 (3520)	1	40-92	<=40	0.52	10
1,4-Dioxane	8270 (3520)	1	22-134	<=40	2.3	10
1,4-Naphthoquinone	8270 (3520)	1	23-49	<=40	0.67	10
1-Naphthylamine	8270 (3520)	1	10-127	<=40	1.2	10
2,3,4,6-Tetrachlorophenol	8270 (3520)	1	10-151	<=40	0.47	10
2,4,5-Trichlorophenol	8270 (3520)	1	62-119	<=40	0.63	10
2,4,6-Trichlorophenol	8270 (3520)	1	61-118	<=40	0.70	10
2,4-Dichlorophenol	8270 (3520)	1	62-112	<=40	0.66	10
2,4-Dimethylphenol	8270 (3520)	1	51-111	<=40	1.0	10
2,4-Dinitrophenol	8270 (3520)	1	13-176	<=40	5.0	50
2,4-Dinitrotoluene (MS)	8270 (3520)	1	45-140	<=40	0.56	10
2,6-Dichlorophenol	8270 (3520)	1	17-125	<=40	0.69	10
2,6-Dinitrotoluene	8270 (3520)	1	65-124	<=40	0.57	10
2-Chloronaphthalene	8270 (3520)	1	58-111	<=40	0.62	10
2-Chlorophenol (MS)	8270 (3520)	1	54-106	<=40	0.72	10
2-Methyl phenol	8270 (3520)	1	57-110	<=40	0.70	10
2-Methylnaphthalene	8270 (3520)	1	51-110	<=40	0.53	10
2-Naphthylamine	8270 (3520)	1	10-119	<=40	0.64	10
2-Nitroaniline	8270 (3520)	1	60-122	<=40	0.69	50
2-Nitrophenol	8270 (3520)	1	59-114	<=40	0.73	10
2-Picoline	8270 (3520)	1	35-102	<=40	0.87	10
3- and 4-Methyl phenol	8270 (3520)	1	49-114	<=40	0.66	10
3,3'-Dichlorobenzidine	8270 (3520)	1	29-101	<=40	1.0	20
3,3'-Dimethylbenzidine	8270 (3520)	1	10-200	<=40	2.5	20
3-Methylcholanthrene	8270 (3520)	1	10-129	<=40	1.4	10
3-Nitroaniline	8270 (3520)	1	46-144	<=40	0.69	50
4,6-Dinitro-2-methylphenol	8270 (3520)	1	42-155	<=40	1.0	50
4-Aminobiphenyl	8270 (3520)	1	20-105	<=40	0.68	10
4-Bromophenyl phenyl ether	8270 (3520)	1	50-112	<=40	0.85	10
4-Chloro-3-methyl-phenol (MS)	8270 (3520)	1	58-118	<=40	0.55	10
4-Chloroaniline	8270 (3520)	1	22-107	<=100	0.52	20
4-Chlorophenylphenyl ether	8270 (3520)	1	59-112	<=40	0.56	10
4-Nitroaniline	8270 (3520)	1	47-127	<=40	0.85	50
4-Nitrophenol (MS)	8270 (3520)	1	30-139	<=40	3.4	50
4-Nitroquinoline-1-oxide	8270 (3520)	1	30-130	<=40	5.0	20

Table 4.3
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Semivolatiles in Groundwater by GC/MS						
5-Nitro-o-toluidine	8270 (3520)	1	10-112	<=40	1.2	10
7,12-Dimethylbenz(a)anthracene	8270 (3520)	1	19-112	<=40	1.3	10
a,a-Dimethylphenethylamine	8270 (3520)	1	10-200	<=40	10	2000
Acenaphthene (MS)	8270 (3520)	1	49-117	<=40	0.64	10
Acenaphthylene	8270 (3520)	1	53-117	<=40	0.58	10
Acetophenone	8270 (3520)	1	54-130	<=40	0.83	10
Aniline	8270 (3520)	1	10-92	<=40	0.65	20
Anthracene	8270 (3520)	1	57-118	<=40	0.88	10
Aramite	8270 (3520)	1	10-168	<=40	1.7	10
Benzo(a)anthracene	8270 (3520)	1	55-119	<=40	0.58	10
Benzo(a)pyrene	8270 (3520)	1	36-128	<=40	1.0	10
Benzo(b)fluoranthene	8270 (3520)	1	44-130	<=40	0.78	10
Benzo(g,h,i)perylene	8270 (3520)	1	45-128	<=40	1.0	10
Benzo(k)fluoranthene	8270 (3520)	1	47-129	<=40	0.73	10
Benzyl alcohol	8270 (3520)	1	54-116	<=40	0.80	10
Bis(2-chloroethoxy) methane	8270 (3520)	1	55-115	<=40	0.78	10
Bis(2-chloroethyl) ether	8270 (3520)	1	48-108	<=40	0.72	10
Bis(2-chloroisopropyl)ether (2,2-Oxybis(1-chloropropane))	8270 (3520)	1	45-114	<=40	0.82	10
Bis(2-ethylhexyl) phthalate	8270 (3520)	1	57-125	<=40	0.98	10
Butyl benzyl phthalate	8270 (3520)	1	62-124	<=40	0.70	10
Carbazole	8270 (3520)	1	60-120	<=40	0.82	10
Chrysene	8270 (3520)	1	56-122	<=40	1.0	10
Diallate	8270 (3520)	1	24-148	<=40	1.6	10
Dibenz(a,h)anthracene	8270 (3520)	1	47-126	<=40	1.0	10
Dibenzofuran	8270 (3520)	1	65-109	<=40	0.69	10
Diethyl phthalate	8270 (3520)	1	61-115	<=40	0.59	10
Dimethoate	8270 (3520)	1	48-77	<=40	0.64	10
Dimethylphthalate	8270 (3520)	1	64-112	<=40	0.59	10
Di-n-butyl phthalate	8270 (3520)	1	58-122	<=40	0.82	10
Di-n-octyl phthalate	8270 (3520)	1	55-128	<=40	0.80	10
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	8270 (3520)	1	10-127	<=40	2.5	10
Diphenylamine/ Nitrosodiphenylamine	8270 (3520)	1	30-130	<=40	0.80	10
Disulfoton	8270 (3520)	1	36-82	<=40	0.78	10
Ethyl methanesulfonate	8270 (3520)	1	45-127	<=40	0.90	10
Ethyl parathion	8270 (3520)	1	10-75	<=40	0.89	10
Famphur	8270 (3520)	1	10-248	<=40	2.2	10
Fluoranthene	8270 (3520)	1	51-125	<=40	1.0	10
Fluorene	8270 (3520)	1	43-124	<=40	0.55	10
Hexachlorobenzene	8270 (3520)	1	60-122	<=40	1.1	10
Hexachlorobutadiene	8270 (3520)	1	43-109	<=40	1.0	10
Hexachlorocyclopentadiene	8270 (3520)	1	1-84	<=100	5.0	10
Hexachloroethane	8270 (3520)	1	35-89	<=40	1.0	10
Hexachlorophene	8270 (3520)	1	1-253	<=100	47	5000
Hexachloropropene	8270 (3520)	1	10-117	<=40	1.2	10
Indeno(1,2,3-cd)pyrene	8270 (3520)	1	40-132	<=40	0.62	10
Isophorone	8270 (3520)	1	60-113	<=40	0.67	10

Table 4.3
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Semivolatiles in Groundwater by GC/MS						
Isosafrole	8270 (3520)	1	26-127	<=40	0.96	10
m-Dinitrobenzene	8270 (3520)	1	10-118	<=40	0.68	10
Methapyrilene	8270 (3520)	1	10-86	<=40	1.9	2000
Methyl parathion	8270 (3520)	1	10-82	<=40	0.78	10
Methylmethanesulfonate	8270 (3520)	1	34-136	<=40	0.85	10
Naphthalene	8270 (3520)	1	42-108	<=40	0.76	10
Nitrobenzene	8270 (3520)	1	57-110	<=40	0.93	10
Nitrosodiphenylamine/ Diphenylamine	8270 (3520)	1	30-130	<=40	0.80	10
N-Nitrosodiethylamine	8270 (3520)	1	50-99	<=40	0.82	10
N-Nitrosodimethylamine	8270 (3520)	1	50-137	<=40	1.1	10
N-Nitrosodi-n-butylamine	8270 (3520)	1	30-123	<=40	0.78	10
N-Nitrosodi-n-propylamine(MS)	8270 (3520)	1	49-135	<=40	0.62	10
N-Nitrosomethylethylamine	8270 (3520)	1	10-279	<=40	2.6	10
N-Nitrosomorpholine	8270 (3520)	1	17-151	<=40	1.1	10
N-Nitrosopiperidine	8270 (3520)	1	21-156	<=40	1.2	10
N-Nitrosopyrrolidine	8270 (3520)	1	52-91	<=40	0.57	10
o,o,o-Triethyl-phosphorothioate	8270 (3520)	1	46-138	<=40	1.3	10
o-Toluidine	8270 (3520)	1	10-129	<=40	0.58	10
p-(Dimethylamino)azobenzene	8270 (3520)	1	10-113	<=40	1.2	10
Pentachlorobenzene	8270 (3520)	1	26-127	<=40	1.0	10
Pentachloronitrobenzene	8270 (3520)	1	10-157	<=40	1.2	10
Pentachlorophenol (MS)	8270 (3520)	1	44-132	<=40	2.5	50
Phenacetin	8270 (3520)	1	18-104	<=40	0.82	10
Phenanthrene	8270 (3520)	1	58-120	<=40	0.74	10
Phenol (MS)	8270 (3520)	1	46-106	<=40	0.99	10
Phorate	8270 (3520)	1	36-113	<=40	0.83	10
p-Phenylenediamine	8270 (3520)	1	10-177	<=40	37	2000
Pronamide	8270 (3520)	1	18-122	<=40	0.72	10
Pyrene(MS)	8270 (3520)	1	49-135	<=40	1.0	10
Pyridine	8270 (3520)	1	10-178	<=40	0.81	50
Safrole	8270 (3520)	1	36-114	<=40	0.99	10
Sulfotepp	8270 (3520)	1	30-117	<=40	0.70	10
Thionazin	8270 (3520)	1	42-93	<=40	0.99	10
Non-Routine Analytes						
Atrazine	8270 (3520)	1	40-132	<=50	0.89	10
Benzaldehyde	8270 (3520)	1	30-150	<=50	1.0	10
1,1-Biphenyl (1,1-Diphenyl)	8270 (3520)	1	53-118	<=50	0.68	10
Caprolactam	8270 (3520)	1	30-150	<=50	0.84	10
3-Nitrophenol	8270 (3520)	1	30-130	<=50	0.78	10

SEVERN TRENT STL

REFERENCES AND NOTES FOR WATER PARAMETERS

A "non-routine analyte" is an analyte that requires advance notice prior to arrival in the laboratory. In general, at least one week notice is required to ensure that the laboratory has sufficient instrument capacity, standards, and reagents to adequately process the samples.

Accuracy data are presented as recoveries for spikes or surrogates. For routine analysis of organics using SW-846 methods, percent recoveries are evaluated only on the subset spike compound lists specified by the methods unless noted in a pre-project plan or QAPP. An (MS) following the parameter name designates the routine matrix and laboratory control spike compounds. Precision data are presented as relative percent difference (% RPD) and are advisory; i.e., not used for laboratory control. Since reportable levels (above detection limit) for most of the organic parameters may not be detected in all environmental samples, precision is usually based on duplicate spike data and evaluated according to method requirements.

Accuracy and precision control limits are primarily derived from in-house laboratory data. Some accuracy and precision limits have been rounded to the nearest "5". In some cases, method limits may be used in lieu of in-house limits because in-house limits are broader than the method limits or are too broad to be usable.

ABBREVIATIONS USED IN THE APPENDICES

PARAMETER-refers to the compound, analyte or measurement being tested; the field of testing

METHOD-refers to the reference method used to measure the parameter

REF-a number designation that corresponds to the method references and citations

ACC-accuracy measured as percent recovery

PREC-precision measured as relative percent difference

RL-reporting limit

MDL-method detection limit

D-analyte detected (meets qualitative identification criteria)

ICP-inductively coupled (argon) plasma

ICP-MS-inductively coupled (argon) plasma coupled to a mass spectrometer

GFAA -graphite furnace atomic absorption

CVAA - cold vapor atomic absorption

IC - ion chromatography

GC/HECD - gas chromatograph equipped with a Hall electrolytic conductivity detector

GC/PID - gas chromatograph equipped with a photoionization detector

GC/FID - gas chromatograph equipped with a flame ionization detector

GC/EC - gas chromatograph equipped with an electron capture detector

GC/NPD - gas chromatograph equipped with a nitrogen-phosphorus detector

GC/MS - gas chromatrograph equipped with a mass spectrometer

HPLC - high performance liquid chromatography

STL

REFERENCES AND NOTES FOR APPENDIX A

REF #	REFERENCE
1	Test Methods for Evaluating Solid Waste, Third Edition, SW-846 (including Update III) USEPA Office of Solid Waste and Emergency Response, Washington, DC.
2	EPA 600/4-79-020:Methods for Chemical Analysis of Water and Wastes; U.S. EPA Office of Research and Development, Cincinatti, OH, March 1983.
3	EPA 600/R-94/111: "Methods for the Determination of Metals in Environmental Samples" May 1994, Supplement 1.
4	Standard Methods for the Examination of Water and Wastewater, 18th Edition, American Public Health Association, Washington, DC.
5	EPA 200.9: Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Adsorption Spectrometry; Revision 1.2, April 1991; EPA-EMSL
6	Laboratory SOP ME72: *Silica: Preparation and Analysis*
7	Laboratory SOP GE115: *Ion Chromatography*
8	Florida Department of Environmental Regulation; Chemistry Laboratory Methods Manual, Tallahassee. "Calculation of Un-ionized Ammonia in Fresh Water" Revision 1, 10/03/83.
9	ASTM Method D 4282-89 "Standard Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion." (reapproved 1994)
10	Method 1650: Absorbable Organic Halides by Adsorption in Microcoulometric Titrations; Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Water and Wastewater: U.S. EPA Office of Water, Engineering, and Analysis Division, Washington, D.C.
11	HACH Method 8141, Hydrazine by p-Dimethylaminobenzaldehyde Method. Adapted from ASTM Manual of Industrial Wastes, D1385-78, 376 (1979)
12	EPA/CE-81-1 Technical Report, May 1981: Environmental Protection Agency/Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material; Procedures for Handling and Chemical Analysis of Sediment and Water Samples.
13	Laboratory SOP GE27: Calculation of Total and Organic Nitrogen
14	Method 1664:N-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons), EPA-821-B-94-004b, April 1995.
15	Method 314.0: Determination of Perchlorate in Drinking Water Using Ion Chromatography, Revision 1.0, November 1999. USEPA Office of Water.
16	American Society for Testing and Materials (ASTM) Method D1498-76 (re-approved in 1981); Laboratory SOP BA72: Oxidation-Reduction Potential (ORP)
17	EPA 600/4-88-039: Methods for the Determination of Organic Compounds in Drinking Water, December, 1988, Revised July, 1991.Methods for the Determination of Organic Compounds in Drinking Water, Supplement I, July 1990. Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, August 1992
18	Code of Federal Regulations, Title 40, Part 136 ; U.S. Government Printing Office: Washington, DC, July 1, 1997.
19	Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater: EPA Method 614, February 1982.
20	Determination of Organophosphorus Pesticides in Wastewater: EPA Method 614.1

SEVERN TRENT STL

REFERENCES AND NOTES FOR APPENDIX A

REF #	REFERENCE
21	Determination of Organophosphorus Pesticides in Industrial and Municipal Wastewater: EPA Method 622 ; January, 1982.
22	Determination of Thiophosphate Pesticides in Industrial and Municipal Wastewater: EPA Method 622.1; January, 1982.
23	Determination of Chlorinated Herbicides in Industrial and Municipal Wastewater. EPA Method 615, January 1982.
24	Method 680: Determination of Pesticides and PCBs in Water and Soil/Sediment by GC/MS U.S. EPA, Washington, D.C..
25	Method 1653: Chlorinated Phenolics in Water by In-situ Acetylation/GC/MS Determination, Method cp. 86.01, National Council of the Paper Industry for Air and Stream Improvement, Inc., 260 Madison Avenue, NY 10016 (July 1986)
26	Method for the Determination of Gasoline Range Organics , State of Tennessee Department of Environment and Conservation, Division of Underground Storage Tanks
27	FL-PRO Method, "Method for Determination of Petroleum Range Organics," FL DEP, Revision 1, November 1, 1995.
28	Method for the Determination of Extractable Petroleum Hydrocarbons by GC/FID ; State of Tennessee Department of Environment and Conservation. Effective May 1, 1999.
29	TNRCC Method for Total Petroleum Hydrocarbons, TNRCC Method 1005 (Revision 03, 6/1/01), State of Texas Method.
30	"NCASI Method DI/HAPS-99.01 Selected HAPS in Condensates by GC/FID. NCASI Southern Regional Center May 2000. National Council for Air and Stream Improvement, Inc., 2000. Methods Manual.
31	"NCASI Method DI/MEOH-94.03 Methanol in Process Liquids by GC/FID" NCASI Southern Regional Center May 2000. National Council for Air and Stream Improvement, Inc., 2000. Methods Manual.
32	RSKSOP 175: Dissolved Oxygen and Methane in Water by GC Headspace Equilibration Technique by Kampbell and Wilson, USEPS, March 1989. Laboratory SOP AR30: *Dissolved Gases in Water*.
33	Method 200.8: Determination of Trace Elements in Water and Wastes by ICP/MS. Revision 5.4 (EMMC Version). USEPA Office of Research and Development (1994)
34	California LUFT (Leaking Underground Fuel Tanks) Method; LUFT Manual: Guidelines for Assessment, Cleanup, and Underground Storage Tank Closure. State of California LUFT Task Force, May 1988.

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	SOIL PARAMETERS					
PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (mg/kg)	RL (mg/kg)
Metals Parameters						
Aluminum	6010 (3050)	1	75-125	<=20	4.5	2.0
Antimony	6010 (3050)	1	75-125	<=20	0.45	2.0
Arsenic	6010 (3050)	1	75-125	<=20	0.67	1.0
Barium	6010 (3050)	1	75-125	<=20	0.30	1.0
Beryllium	6010 (3050)	1	75-125	<=20	0.017	0.40
Boron	6010 (3050)	1	75-125	<=20	1.30	5.0
Cadmium	6010 (3050)	1	75-125	<=20	0.22	0.50
Calcium	6010 (3050)	1	75-125	<=20	2.4	50
Chromium	6010 (3050)	1	75-125	<=20	0.13	1.0
Cobalt	6010 (3050)	1	75-125	<=20	0.17	1.0
Copper	6010 (3050)	1	75-125	<=20	0.17	2.0
Iron	6010 (3050)	1	75-125	<=20	4.2	5.0
Lead	6010 (3050)	1	75-125	<=20	0.21	0.50
Magnesium	6010 (3050)	1	75-125	<=20	1.2	50
Manganese	6010 (3050)	1	75-125	<=20	0.21	1.0
Molybdenum	6010 (3050)	1	75-125	<=20	0.33	1.0
Nickel	6010 (3050)	1	75-125	<=20	0.26	4.0
Potassium	6010 (3050)	1	75-125	<=20	1.3	100
Selenium	6010 (3050)	1	75-125	<=20	0.90	2.5
Silver	6010 (3050)	1	75-125	<=20	0.099	1.0
Sodium	6010 (3050)	1	75-125	<=20	50	100
Strontium	6010 (3050)	1	75-125	<=20	0.15	1.0
Thallium	6010 (3050)	1	75-125	<=20	1.30	2.5
Tin	6010 (3050)	1	75-125	<=20	4.0	10
Titanium	6010 (3050)	1	70-130	<=20	0.040	1.0
Vanadium	6010 (3050)	1	75-125	<=20	0.14	1.0
Zinc	6010 (3050)	1	75-125	<=20	0.75	2.0
Mercury	7471	1	80-120	<=20	0.0040	0.020
General Chemistry Parameters						
Ammonia (as N)	350.1(EPA-CE:3-140)	3/4	75-125	<=30	0.075	0.15
Polychlorinated Biphenyls as Aroclors by GC/EC (Sonication Extraction)						
PCB-1016	8082 (3550)	1	34-128	<=50	6.7	33
PCB 1221	8082 (3550)	1	30-130	<=50	6.8	67
PCB 1232	8082 (3550)	1	30-130	<=50	6.2	33
PCB-1242	8082 (3550)	1	30-130	<=50	7.5	33
PCB-1248	8082 (3550)	1	30-130	<=50	8.0	33
PCB-1254	8082 (3550)	1	30-130	<=50	5.2	33
PCB-1260	8082 (3550)	1	28-168	<=51	6.4	33
PCB-1268	8082 (3550)	1	30-150	<=52	11	33
Surrogates						
2,4,5,6-Tetrachloro-m-xylene	8082 (3550)	1	30-150	NA	NA	NA
Decachlorobiphenyl	8082 (3550)	1	30-150	NA	NA	NA

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	SOIL PARAMETERS					
PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (mg/kg)	RL (mg/kg)
Chlorinated Pesticides by GC/EC (Sonication Extraction)						
4,4'-DDD	8081 (3550)	1	35-149	<=50	0.30	3.3
4,4'-DDE	8081 (3550)	1	35-122	<=50	0.30	3.3
4,4'-DDT (MS)	8081 (3550)	1	24-171	<=50	0.27	3.3
Aldrin (MS)	8081 (3550)	1	26-139	<=50	0.14	1.7
alpha Chlordane	8081 (3550)	1	42-125	<=50	0.17	1.7
alpha-BHC	8081 (3550)	1	23-127	<=50	0.52	1.7
beta-BHC	8081 (3550)	1	22-134	<=50	0.47	1.7
Chlorobenzilate	8081 (3550)	1	73-201	<=50	3.8	17
delta-BHC	8081 (3550)	1	43-123	<=50	0.23	1.7
Dieldrin (MS)	8081 (3550)	1	29-146	<=50	0.35	3.3
Endosulfan I	8081 (3550)	1	31-124	<=50	0.16	1.7
Endosulfan II	8081 (3550)	1	31-127	<=50	0.27	3.3
Endosulfan sulfate	8081 (3550)	1	55-136	<=50	0.37	3.3
Endrin (MS)	8081 (3550)	1	45-148	<=50	0.32	3.3
Endrin aldehyde	8081 (3550)	1	36-123	<=50	0.65	3.3
Endrin ketone	8081 (3550)	1	47-156	<=50	0.32	3.3
gamma Chlordane	8081 (3550)	1	37-152	<=50	0.22	1.7
gamma-BHC (Lindane) (MS)	8081 (3550)	1	16-144	<=50	0.14	1.7
Heptachlor (MS)	8081 (3550)	1	19-150	<=50	0.32	1.7
Heptachlor epoxide	8081 (3550)	1	43-132	<=50	0.21	1.7
Isodrin	8081 (3550)	1	14-188	<=50	0.33	3.3
Kepone	8081 (3550)	1	10-65	<=50	6.7	170
Methoxychlor	8081 (3550)	1	13-208	<=50	0.47	17
Technical Chlordane	8081 (3550)	1	41-177	<=50	3	17
Toxaphene	8081 (3550)	1	36-159	<=50	12	170
Surrogates						
Tetrachloro-m-xylene	8081 (3550)	1	30-150	NA	NA	NA
Decachlorobiphenyl	8081 (3550)	1	30-150	NA	NA	NA
Non-routine Compounds						
2,4'-DDD	8081 (3550)	1	35-149	<=50	3.3	3.3
2,4'-DDE	8081 (3550)	1	35-122	<=50	3.3	3.3
2,4'-DDT	8081 (3550)	1	24-171	<=50	3.3	3.3
Chlorinated Herbicides by GC/EC						
2,4-D (MS)	8151	1	53-135	<=50	0.97	8.3
2,4-DB	8151	1	31-106	<=50	1.0	8.3
2,4,5-T (MS)	8151	1	43-121	<=50	0.56	8.3
2,4,5-TP (Silvex) (MS)	8151	1	38-119	<=50	0.52	8.3
Dalapon	8151	1	59-164	<=50	10	330
Dicamba	8151	1	59-107	<=50	1.5	8.3
Dichlorprop	8151	1	48-96	<=50	1.8	8.3
Dinoseb	8151	1	31-92	<=50	14	100
MCPA	8151	1	57-117	<=50	42	2000
MCPP	8151	1	30-140	<=50	220	2000
Pentachlorophenol	8151	1	71-109	<=50	0.98	8.3
Surrogates						
2,4-Dichlorophenyl acetic acid	8151	1	34-127	NA	NA	NA
Non-Routine Compounds						
Picloram	8151	1	30-150	<=50	2.6	8.3

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	SOIL PARAMETERS					
PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (mg/kg)	RL (mg/kg)
Volatiles by GC/MS						
1,1,1,2-Tetrachloroethane	8260 (5035)	1	70-130	<=50	0.45	5.0
1,1,1-Trichloroethane	8260 (5035)	1	58-139	<=50	0.50	5.0
1,1,2,2-Tetrachloroethane	8260 (5035)	1	64-130	<=50	0.60	5.0
1,1,2-Trichloro-1,2,2-trifluoroethane	8260 (5035)	1	70-130	<=50	1.2	5.0
1,1,2-Trichloroethane	8260 (5035)	1	76-120	<=50	0.54	5.0
1,1-Dichloroethane	8260 (5035)	1	43-157	<=50	0.4	5.0
1,1-Dichloroethene (MS)	8260 (5035)	1	52-143	<=50	0.56	5.0
1,1-Dichloropropene	8260 (5035)	1	76-126	<=50	0.64	5.0
1,2,3-Trichlorobenzene	8260 (5035)	1	29-169	<=50	0.38	5.0
1,2,3-Trichloropropane	8260 (5035)	1	33-210	<=50	0.87	5.0
1,2,4-Trichlorobenzene	8260 (5035)	1	49-152	<=50	0.46	5.0
1,2,4-Trimethylbenzene	8260 (5035)	1	74-133	<=50	0.4	5.0
1,2-Dibromo-3-chloropropane (DBCP)	8260 (5035)	1	21-180	<=100	1.0	10
1,2-Dibromoethane (EDB)	8260 (5035)	1	76-130	<=50	0.73	5.0
1,2-Dichlorobenzene	8260 (5035)	1	81-122	<=50	0.53	5.0
1,2-Dichloroethane	8260 (5035)	1	65-133	<=50	0.59	5.0
1,2-Dichloroethenes (total)	8260 (5035)	1	35-154	<=50	0.62	10
1,2-Dichloropropane	8260 (5035)	1	77-118	<=50	0.42	5.0
1,3,5-Trimethylbenzene	8260 (5035)	1	72-124	<=50	0.34	5.0
1,3-Dichlorobenzene	8260 (5035)	1	84-120	<=50	0.5	5.0
1,3-Dichloropropane	8260 (5035)	1	73-146	<=50	0.56	5.0
1,4-Dichlorobenzene	8260 (5035)	1	67-133	<=50	0.47	5.0
2,2-Dichloropropane	8260 (5035)	1	28-187	<=50	0.59	5.0
2-Butanone (Methyl Ethyl Ketone-MEK)	8260 (5035)	1	30-149	<=50	1.8	25
2-Chloro-1,3-butadiene (Chloroprene)	8260 (5035)	1	70-130	<=50	2.1	5.0
2-Chloroethyl vinyl ether	8260 (5035)	1	D-200	<=100	50.0	50
2-Chlorotoluene	8260 (5035)	1	49-219	<=50	0.5	5.0
2-Hexanone	8260 (5035)	1	30-148	<=50	2.0	25
3-Chloropropene (Allyl chloride)	8260 (5035)	1	40-165	<=50	2.3	5.0
4-Chlorotoluene	8260 (5035)	1	45-218	<=50	0.57	5.0
4-Methyl-2-pentanone (MIBK)	8260 (5035)	1	29-150	<=50	1.9	25
Acetone	8260 (5035)	1	28-143	<=100	2.6	50
Acetonitrile	8260 (5035)	1	61-148	<=50	53	200
Acrolein	8260 (5035)	1	1-123	<=100	23	100
Acrylonitrile	8260 (5035)	1	44-125	<=50	16	100
Benzene (MS)	8260 (5035)	1	79-118	<=50	0.52	5.0
Bromobenzene	8260 (5035)	1	77-147	<=50	0.4	5.0
Bromochloromethane	8260 (5035)	1	63-136	<=50	0.47	5.0
Bromodichloromethane	8260 (5035)	1	74-128	<=50	0.42	5.0
Bromoform	8260 (5035)	1	62-137	<=50	0.62	5.0
Bromomethane	8260 (5035)	1	26-160	<=100	4.3	5.0
Carbon disulfide	8260 (5035)	1	32-157	<=50	0.4	5.0
Carbon tetrachloride	8260 (5035)	1	62-140	<=50	0.36	5.0
Chlorobenzene (MS)	8260 (5035)	1	81-120	<=50	0.84	5.0
Chloroethane	8260 (5035)	1	20-140	<=100	1.0	5.0
Chloroform	8260 (5035)	1	77-125	<=50	0.36	5.0
Chloromethane	8260 (5035)	1	42-140	<=100	0.42	5.0
cis-1,2-Dichloroethene	8260 (5035)	1	69-131	<=50	0.30	5.0
cis-1,3-Dichloropropene	8260 (5035)	1	71-123	<=50	0.45	5.0
Dibromochloromethane	8260 (5035)	1	67-135	<=50	0.5	5.0
Dibromomethane	8260 (5035)	1	71-134	<=50	0.42	5.0

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	SOIL PARAMETERS					
PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (mg/kg)	RL (mg/kg)
Volatiles by GC/MS						
Dichlorodifluoromethane	8260 (5035)	1	70-130	<=100	0.90	5.0
Ethyl methacrylate	8260 (5035)	1	66-152	<=50	1.2	5.0
Ethylbenzene	8260 (5035)	1	82-118	<=50	0.6	5.0
Hexachlorobutadiene	8260 (5035)	1	29-135	<=100	0.68	5.0
Iodomethane	8260 (5035)	1	35-162	<=50	1.3	5.0
Isobutyl alcohol	8260 (5035)	1	74-136	<=50	83	200
Isopropylbenzene	8260 (5035)	1	75-134	<=50	0.35	5.0
m&p-Xylene	8260 (5035)	1	74-121	<=50	1.5	5.0
Methacrylonitrile	8260 (5035)	1	60-142	<=50	31	100
Methyl methacrylate	8260 (5035)	1	54-155	<=50	2.0	5.0
Methyl t-butyl ether (MTBE)	8260 (5035)	1	37-168	<=50	0.69	50
Methylene chloride	8260 (5035)	1	54-150	<=100	0.87	5.0
Naphthalene	8260 (5035)	1	42-250	<=50	1.8	5.0
n-Butylbenzene	8260 (5035)	1	59-120	<=50	0.42	5.0
n-Propylbenzene	8260 (5035)	1	67-134	<=50	0.47	5.0
o-Xylene	8260 (5035)	1	74-122	<=50	0.44	5.0
Pentachloroethane	8260 (5035)	1	1-195	<=100	1.4	25
p-Isopropyltoluene	8260 (5035)	1	39-141	<=50	0.39	5.0
Propionitrile (ethylcyanide)	8260 (5035)	1	58-142	<=50	30	100
sec-Butylbenzene	8260 (5035)	1	60-128	<=50	0.45	5.0
Styrene	8260 (5035)	1	80-118	<=50	0.53	5.0
tert-Butylbenzene	8260 (5035)	1	62-140	<=50	0.52	5.0
Tetrachloroethene	8260 (5035)	1	79-132	<=50	0.91	5.0
Toluene (MS)	8260 (5035)	1	80-118	<=50	0.69	5.0
trans-1,2-Dichloroethene	8260 (5035)	1	35-154	<=50	0.37	5.0
trans-1,3-Dichloropropene	8260 (5035)	1	75-126	<=50	0.45	5.0
trans-1,4-Dichloro-2-butene	8260 (5035)	1	27-150	<=100	3.6	10
Trichloroethene (MS)	8260 (5035)	1	80-122	<=50	4.4	5.0
Trichlorofluoromethane	8260 (5035)	1	38-146	<=100	0.4	5.0
Vinyl acetate	8260 (5035)	1	1-184	<=100	0.83	10
Vinyl chloride	8260 (5035)	1	34-154	<=100	0.59	5.0
Xylenes (total)	8260 (5035)	1	74-122	<=50	1.9	10
Surrogates						
p-Bromofluorobenzene	8260 (5035)	1	68-121	NA	NA	NA
Dibromofluoromethane	8260 (5035)	1	66-127	NA	NA	NA
Toluene-d8	8260 (5035)	1	65-128	NA	NA	NA
Non-Routine Compounds						
1-Chlorohexane	8260 (5035)	1	70-130	<=50	0.72	5.0
Cyclohexane	8260 (5035)	1	70-130	<=50	10	10
Cyclohexanone	8260 (5035)	1	70-130	<=50	15	50
Diethyl ether	8260 (5035)	1	70-130	<=50	0.78	10
Furan	8260 (5035)	1	70-130	<=50	0.36	5.0
Methyl acetate	8260 (5035)	1	70-130	<=50	1.6	10
Methyl cyclohexane	8260 (5035)	1	70-130	<=50	0.48	10
Tetrahydrofuran	8260 (5035)	1	70-130	<=50	0.36	10
Semivolatiles (Base-Neutrals/Acids) by GC/MS (Sonication Extraction)						
1,2,4,5-Tetrachlorobenzene	8270 (3550)	1	37-124	<=50	31	330
1,2,4-Trichlorobenzene (MS)	8270 (3550)	1	36-98	<=50	21	330
1,2-Dichlorobenzene	8270 (3550)	1	35-93	<=50	23	330

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	SOIL PARAMETERS					
PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (mg/kg)	RL (mg/kg)
Semivolatiles (Base-Neutrals/Acids) by GC/MS (Sonication Extraction)						
1,2-Diphenyl hydrazine	8270 (3550)	1	22-129	<=50	22	330
1,3,5-Trinitrobenzene	8270 (3550)	1	1-131	<=100	82	330
1,3-Dichlorobenzene	8270 (3550)	1	34-90	<=50	26	330
1,4-Dichlorobenzene (MS)	8270 (3550)	1	32-90	<=50	22	330
1,4-Dioxane	8270 (3550)	1	1-156	<=100	66	330
1,4-naphthoquinone	8270 (3550)	1	1-122	<=100	39	330
1-Methylnaphthalene	8270 (3550)	1	12-128	<=50	29	330
1-Naphthylamine	8270 (3550)	1	1-47	<=100	82	330
2,4,5-Trichlorophenol	8270 (3550)	1	46-116	<=50	32	330
2,4,6-Trichlorophenol	8270 (3550)	1	44-113	<=50	20	330
2,4-Dichlorophenol	8270 (3550)	1	43-108	<=50	23	330
2,4-Dimethylphenol	8270 (3550)	1	40-112	<=50	35	330
2,4-Dinitrophenol	8270 (3550)	1	1-131	<=50	170	1700
2,4-Dinitrotoluene (MS)	8270 (3550)	1	32-128	<=50	19	330
2,6-Dichlorophenol	8270 (3550)	1	20-138	<=50	53	330
2,6-Dinitrotoluene	8270 (3550)	1	38-128	<=50	19	330
2-Acetylaminofluorene	8270 (3550)	1	1-126	<=100	38	330
2-Chloronaphthalene	8270 (3550)	1	41-110	<=50	24	330
2-Chlorophenol (MS)	8270 (3550)	1	36-99	<=50	27	330
2-Methyl phenol (o-Cresol)	8270 (3550)	1	38-107	<=50	30	330
2-Methylnaphthalene	8270 (3550)	1	39-104	<=50	24	330
2-Naphthylamine	8270 (3550)	1	1-51	<=100	82	330
2-Nitroaniline	8270 (3550)	1	38-124	<=50	23	1700
2-Nitrophenol	8270 (3550)	1	38-104	<=50	20	330
2-Picoline	8270 (3550)	1	19-76	<=50	82	330
3- and 4-Methyl phenol	8270 (3550)	1	37-106	<=50	29	330
3,3'-Dichlorobenzidine	8270 (3550)	1	1-118	<=50	420	660
3,3'-Dimethylbenzidine	8270 (3550)	1	1-58	<=100	30	1700
3-Methylcholanthrene	8270 (3550)	1	1-151	<=100	26	330
3-Nitroanline	8270 (3550)	1	19-118	<=50	33	1700
4,6-Dinitro-2-methylphenol	8270 (3550)	1	11-142	<=50	200	1700
4-Aminobiphenyl	8270 (3550)	1	10-47	<=50	54	330
4-Bromophenyl phenyl ether	8270 (3550)	1	38-106	<=50	31	330
4-Chloro-3-methylphenol (MS)	8270 (3550)	1	39-113	<=50	34	330
4-Chloroaniline	8270 (3550)	1	7-103	<=50	26	660
4-Chlorophenylphenyl ether	8270 (3550)	1	42-111	<=50	19	330
4-Nitroaniline	8270 (3550)	1	32-130	<=50	17	1700
4-Nitrophenol (MS)	8270 (3550)	1	21-132	<=50	210	1700
4-Nitroquinoline-1-oxide	8270 (3550)	1	1-200	<=100	166	3300
7,12-Dimethylbenz(a)anthracene	8270 (3550)	1	11-128	<=50	31	330
a,a-Dimethylphenethylamine	8270 (3550)	1	1-65	<=100	490	6700
Acenaphthene (MS)	8270 (3550)	1	36-108	<=50	19	330
Acenaphthylene	8270 (3550)	1	41-112	<=50	17	330
Acetophenone	8270 (3550)	1	24-108	<=50	22	330
Aniline	8270 (3550)	1	1-86	<=100	21	330
Anthracene	8270 (3550)	1	46-115	<=50	23	330
Aramite	8270 (3550)	1	1-140	<=100	60	330
Benzidine	8270 (3550)	1	1-95	<=100	83	2700
Benzo(a)anthracene	8270 (3550)	1	46-116	<=50	31	330
Benzo(a)pyrene	8270 (3550)	1	37-120	<=50	19	330
Benzo(b)fluoranthene	8270 (3550)	1	35-122	<=50	26	330

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	SOIL PARAMETERS					
PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (mg/kg)	RL (mg/kg)
Semivolatiles (Base-Neutrals/Acids) by GC/MS (Sonication Extraction)						
Benzo(g,h,i)perylene	8270 (3550)	1	41-122	<=50	23	330
Benzo(k)fluoranthene	8270 (3550)	1	36-124	<=50	36	330
Benzoic acid	8270 (3550)	1	10-94	<=100	170	1700
Benzyl alcohol	8270 (3550)	1	30-98	<=50	38	330
Bis(2-chloroethoxy) methane	8270 (3550)	1	38-106	<=50	24	330
Bis(2-chloroethyl) ether	8270 (3550)	1	30-98	<=50	28	330
Bis(2-chloroisopropyl) ether	8270 (3550)	1	16-116	<=50	35	330
Bis(2-ethylhexyl) phthalate	8270 (3550)	1	25-134	<=50	38	330
Butyl benzyl phthalate	8270 (3550)	1	43-127	<=50	27	330
Carbazole	8270 (3550)	1	47-118	<=50	28	330
Chrysene	8270 (3550)	1	46-118	<=50	25	330
Diallate	8270 (3550)	1	24-137	<=50	56	330
Dibenz(a,h)anthracene	8270 (3550)	1	41-124	<=50	24	330
Dibenzofuran	8270 (3550)	1	44-108	<=50	19	330
Diethylphthalate	8270 (3550)	1	41-118	<=50	22	330
Dimethoate	8270 (3550)	1	22-102	<=50	45	330
Dimethylphthalate	8270 (3550)	1	43-114	<=50	19	330
Di-n-butylphthalate	8270 (3550)	1	35-93	<=50	28	330
Di-n-octylphthalate	8270 (3550)	1	43-129	<=50	31	330
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	8270 (3550)	1	1-114	<=100	83	330
Diphenylamine/ N-nitrosodiphenylamine	8270 (3550)	1	16-113	<=50	23	330
Disulfoton	8270 (3550)	1	31-65	<=50	55	330
Ethyl methanesulfonate	8270 (3550)	1	28-113	<=50	66	330
Ethyl parathion	8270 (3550)	1	28-113	<=50	55	330
Famphur	8270 (3550)	1	1-124	<=100	98	330
Fluoranthene	8270 (3550)	1	41-124	<=50	26	330
Fluorene	8270 (3550)	1	37-113	<=50	22	330
Hexachlorobenzene	8270 (3550)	1	46-115	<=50	26	330
Hexachlorobutadiene	8270 (3550)	1	42-105	<=50	20	330
Hexachlorocyclopentadiene	8270 (3550)	1	20-109	<=50	83	330
Hexachloroethane	8270 (3550)	1	31-88	<=50	20	330
Hexachlorophene	8270 (3550)	1	1-164	<=100	12500	170000
Hexachloropropene	8270 (3550)	1	34-116	<=50	45	330
Indeno(1,2,3-cd)pyrene	8270 (3550)	1	36-133	<=50	26	330
Isophorone	8270 (3550)	1	37-106	<=50	22	330
Isosafrole	8270 (3550)	1	25-133	<=50	64	330
m-Dinitrobenzene	8270 (3550)	1	1-134	<=100	50	330
Methapyrilene	8270 (3550)	1	1-110	<=100	42	67000
Methyl methanesulfonate	8270 (3550)	1	37-108	<=50	55	330
Methyl parathion	8270 (3550)	1	19-60	<=50	47	330
Naphthalene	8270 (3550)	1	34-97	<=50	19	330
Nitrobenzene	8270 (3550)	1	33-106	<=50	35	330
N-Nitro-o-toluidine	8270 (3550)	1	10-100	<=50	32	330
N-Nitrosodiethylamine	8270 (3550)	1	10-111	<=50	44	330
N-Nitrosodimethylamine	8270 (3550)	1	10-132	<=50	59	330
N-Nitroso-di-N-butylamine	8270 (3550)	1	32-99	<=50	52	330
N-Nitrosodiphenylamine/ Diphenylamine	8270 (3550)	1	16-113	<=50	23	330
N-Nitrosomethylethylamine	8270 (3550)	1	22-137	<=50	52	330
N-Nitrosomorpholine	8270 (3550)	1	18-129	<=50	66	330
N-Nitrosopiperidine	8270 (3550)	1	26-125	<=50	71	330
N-Nitrosopyrrolidine	8270 (3550)	1	20-108	<=50	45	330

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	SOIL PARAMETERS					
PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (mg/kg)	RL (mg/kg)
Semivolatiles (Base-Neutrals/Acids) by GC/MS (Sonication Extraction)						
N-Nitrosos-di-N-propylamine (MS)	8270 (3550)	1	24-108	<=50	28	330
o,o,o-Triethylphosphorothioate	8270 (3550)	1	28-124	<=50	72	330
o-Toluidine	8270 (3550)	1	10-58	<=50	56	330
p-(Dimethylamino)azobenzene	8270 (3550)	1	1-124	<=100	38	330
Pentachlorobenzene	8270 (3550)	1	30-133	<=50	64	330
Pentachloronitrobenzene	8270 (3550)	1	22-127	<=50	56	330
Pentachlorophenol (MS)	8270 (3550)	1	27-116	<=50	83	1700
Phenacetin	8270 (3550)	1	1-117	<=100	51	330
Phenanthrene	8270 (3550)	1	47-114	<=50	29	330
Phenol (MS)	8270 (3550)	1	34-98	<=50	30	330
Phorate	8270 (3550)	1	37-81	<=50	69	330
p-Phenylenediamine	8270 (3550)	1	1-130	<=100	350	1700
Pronamide	8270 (3550)	1	27-84	<=50	63	330
Pyrene (MS)	8270 (3550)	1	36-128	<=50	20	330
Pyridine	8270 (3550)	1	1-107	<=100	30	330
Safrole	8270 (3550)	1	34-119	<=50	53	330
Sulfotepp	8270 (3550)	1	31-102	<=50	55	330
Tetrachlorophenols (2,3,4,5 + 2,3,4,6)	8270 (3550)	1	21-106	<=50	45	330
Thionazin	8270 (3550)	1	21-86	<=50	42	330
Surrogates						
2-Fluorobiphenyl	8270 (3550)	1	38-104	NA	NA	NA
2-Fluorophenol	8270 (3550)	1	36-101	NA	NA	NA
Nitrobenzene-d5	8270 (3550)	1	33-94	NA	NA	NA
Phenol-d5	8270 (3550)	1	38-102	NA	NA	NA
p-Terphenyl-d14	8270 (3550)	1	40-129	NA	NA	NA
2,4,6- Tribromophenol	8270 (3550)	1	27-124	NA	NA	NA
Notes:						
1) Shadded chemicals indicate the chemical is not included in the Superfund Target Compound or Analyte List.						
2) NA = Not Applicable.						

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REFERENCES AND NOTES FOR SOIL PARAMETERS

A "non-routine analyte" is an analyte that requires advance notice prior to arrival in the laboratory. In general, at least one week notice is required to ensure that the laboratory has sufficient instrument capacity, standards, and reagents to adequately process the samples.

Accuracy data are presented as recoveries for spikes or surrogates. For routine analysis of organics using SW-846 methods, percent recoveries are evaluated only on the subset spike compound lists specified by the methods unless noted in a pre-project plan or QAPP. An (MS) following the parameter name designates the routine matrix and laboratory control spike compounds. Precision data are presented as relative percent difference (% RPD) and are advisory; i.e., not used for laboratory control. Since reportable levels (above detection limit) for most of the organic parameters may not be detected in all environmental samples, precision is usually based on duplicate spike data and evaluated according to method requirements.

Accuracy and precision control limits are primarily derived from in-house laboratory data. Some accuracy and precision limits have been rounded to the nearest "5". In some cases, method limits may be used in lieu of in-house limits because in-house limits are broader than the method limits or are too broad to be usable.

ABBREVIATIONS USED IN THE APPENDIX

PARAMETER-refers to the compound, analyte, or measurement being tested or performed (the field of test)

METHOD-refers to the reference method used to measure the parameter

REF-a number designation that corresponds to the method references and citations listed below

ACC-accuracy control limits measured as percent recovery

PREC-precision measured as relative percent difference

RL-reporting limit

MDL-method detection limit

D-analyte detected (meets qualitative identification criteria)

ICP-inductively coupled (argon) plasma

ICP-inductively coupled (argon) plasma coupled to a mass spectrometer

GFAA -graphite furnace atomic absorption

CVAA - cold vapor atomic absorption

IC - ion chromatography

GC/HECD - gas chromatograph equipped with a Hall electrolytic conductivity detector

GC/PID- gas chromatograph equipped with a photoionization detector

GC/FID - gas chromatograph equipped with a flame ionization detector

GC/EC - gas chromatograph equipped with an electron capture detector

GC/NPD - gas chromatograph equipped with a nitrogen-phosphorus detector

GC/MS - gas chromatrograph equipped with a mass spectrometer

HPLC -high performance liquid chromatography

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REFERENCES AND NOTES FOR SOIL PARAMETERS

REF #	REFERENCE
1	Test Methods for Evaluating Solid Waste, Third Edition, SW-846 (including Update III) USEPA Office of Solid Waste and Emergency Response, Washington, DC.
2	ASTM 3987-85: Standard Test Method for Shake Extraction of Solid Waste with Water. American Society of Testing and Materials(1992).
3	EPA 600/4-79-020:Methods for Chemical Analysis of Water and Wastes; U.S. EPA Office of Reseach and Development, Cincinnati, OH, March 1983.
4	EPA/CE-81-1 Technical Report, May 1981: Environmental Protection Agency/Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material; Procedures for Handling and Chemical Analysis of Sediment and Water Samples.
5	ASTM D240-87; Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter. American Society of Testing and Materials (1991)
6	Methods of Soil Analysis, American Society of Agronomy, Inc., Number 9, Part 2, page 570, (Walkley-Black Procedure).
7	Determination of Total Organic Carbon in Sediment; USEPA Region II Environmental Services Division Monitoring Management Branch; Edison, NJ.Lloyd Kahn, QA Specialist. 7/10/86
8	Methods for the Determination of Inorganic Substances in Environmental Samples, USEPA Office of Research and Development: Washington, DC August 1993, EPA/600/R-93/100.
9	Method 1664:N-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons) EPA-821-B-94-004b, April 1995.
10	Method 314.0: Determination of Perchlorate in Drinking Water Using Ion Chromatography, revision 1.0, November 1999. USEPA Office of Groundwater and Drinking Water.
11	Development of an Analytical Method of the Determination of Acid Volatile Sulfides (AVS) in Sediments; EPA Contract No. 68-03-3534, October 1990.
12	EPA/600/8-78/017: Microbiological Methods for Monitoring the Environment - Water and Wastes, December 1978.
13	Method for the Determination of Gasoline Range Organics , State of Tennessee Department of Environment and Conservation, Division of Underground Storage Tanks
14	FL-PRO Method, "Method for Determination of Petroleum Range Organics," FL DEP, Revision 1, November 1, 1995.
15	Method for the Determination of Extractable Petroleum Hydrocarbons by GC/FID ; State of Tennessee Department of Environment and Conservation. Effective May 1, 1999.
16	TNRCC Method for Total Petroleum Hydrocarbons, TNRCC Method 1005 (Revision 03, 6/1/01), State of Texas Method.
17	Method 680: Determination of Pesticides and PCBs in Water and Soils/Sediment by Gas Chromatography/Mass Spectrometry. November 1985. Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, USEPA, Cincinnati, OH
18	Test Methods for Evaluating Solid Waste, Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC,. Method 8280
19	California LUFT (Leaking Underground Fuel Tanks) Method; LUFT Manual: Guidelines for Assessment, Cleanup, and Underground Storage Tank Closure. State of California LUFT Task Force, May 1988.

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) outlines the procedures used in the redevelopment of wells. The purpose of well development is to remove well drilling fluids, solids, or other particulates that may have been introduced into the formation and/or well or deposited on the boring wall in a recently installed well during drilling and construction activities. This restores the hydraulic conductivity of the aquifer material surrounding the well to near pre-well installation conditions. Properly developed monitoring wells allow for the collection of ground water samples which are chemically and physically representative of the aquifer of concern. The procedure is also applicable to older or improperly developed wells that are suspected of not providing representative groundwater samples. Monitoring wells will be developed by pumping or bailing.

2.0 EQUIPMENT NEEDED

The following items are required to properly develop groundwater monitoring wells:

- Well keys
- Electronic water level indicator
- Calculator
- Field notebook
- Waterproof pen
- Submersible pump (or other appropriate pump), air-lift system or PVC, Teflon or stainless steel bailer (sized appropriately for well)
- Nylon rope or wireline (for deep wells) for bailing
- Surge block (sized appropriately for well)
- PVC or stainless steel pipe for operating surge block (sized appropriately for well)
- Thermometer
- pH meter (with automatic temperature compensation)
- Calibration pH buffer solutions
- Conductivity meter
- Polyethylene or glass container (for field parameter measurements)
- Plastic squeeze bottle filled with deionized water
- 5-gallon bucket
- Drums or other large container for redevelopment water, if required
- Appropriate health and safety equipment as specified in the Health and Safety Plan
- Appropriate decontamination equipment as specified in the decontamination SOP
- Well completion information

- Plastic sheeting, if required
- Generator or 12 volt battery if electric pump is to be used for developing

3.0 PROCEDURES

WELL REDEVELOPMENT PROCEDURE

Well Redevelopment: URS personnel will direct a program for the redevelopment of the well without the use of dispersing agents, acids, or explosives. The objectives of well redevelopment are to: (a) assure that groundwater enters the well screen freely, thus yielding a representative groundwater sample and an accurate water level measurement; (b) remove all water that may have been introduced during drilling and well installation; (c) remove very fine-grained sediment in the filter pack and nearby formation so that groundwater samples are not highly turbid and so that silting of the well does not occur. Redevelopment will consist of surging and bailing or pumping until little or no sediment enters the well. Each monitoring well will be redeveloped until a minimum of five well volumes have been removed and pH, specific conductance, and temperature readings stabilize within 10% over a minimum of two successive readings.

If the addition of water is required to facilitate surging and bailing, only formation water from that well will be used. If this is not practical due to tightness of the formation then only bailing will be performed. In all cases, care will be taken not to collapse well screens during redevelopment activities.

The volume of water required for removal during redevelopment is calculated using the following method:

1. Measure the depth to water in the well from the measuring point. This is usually a notched point on the top of PVC riser pipe which has been surveyed.
2. Measure the total depth of the well from the same measuring point used for measuring the depth to water.
3. Calculate the height of water in the well casing by subtracting the depth of water from the total well depth.
4. Calculate the number of gallons of water corresponding to one well volume. This is done by multiplying the height of water in the well casing by the conversion factor corresponding to the inside diameter of the well casing.

The following equation shall be used to calculate the volume of water to be removed during well evacuation:

a) Volume of water in casing (gal) =
[ ]*water column (ft)*π*(well radius)^2*0.0043

b) Volume of water in annulus (gal) =

[ ]*water column (ft)*π*[(borehole radius)^2-(well radius)^2*0.0043*0.3

Multiply the volume of one well casing volume, plus the annulus volume if required, by [ ] to obtain the minimum volume of water to be evacuated.

During the well redevelopment activities, field measurements of temperature, pH, and specific conductivity are made, and the clarity, color, any presence of odors, and other comments regarding water quality are noted in the field notebook and on the well development log. The date, time, and volume of water removed is also recorded at this time. Measurements of pH, conductivity and temperature along with observations will be recorded as each well volume of water is removed. A sample of water will be collected for measurement of pH, conductivity, and temperature at the beginning of well redevelopment in order to establish a baseline for comparison with the water quality as well redevelopment proceeds.

3.0 DOCUMENTATION

Documentation of observations and data acquired in the field will provide information on well redevelopment and also provide a permanent record. These observations and data will be recorded with waterproof ink in a bound weatherproof field book with consecutively numbered pages and on the well redevelopment form attached.

As part of the redevelopment process, the following information will be recorded in the field book:

- Date
- Time
- Weather
- Personnel
- Well designation
- Well location
- Date(s) and time of well redevelopment
- Static water level from top of well casing before and after redevelopment
- Volume of water in well prior to redevelopment
- Volume of water removed and time of removal
- Depth from top of well casing to bottom of well
- Screen length
- Depth from top of well casing to top of sediment inside well, if present, before and after redevelopment

- Field measurements of pH, conductivity, and temperature taken during and after redevelopment
- Physical character of removed water throughout redevelopment (color, odor, and turbidity)
- Type and size/capacity of pump and/or bailer
- Description of redevelopment technique
- Decontamination observations
- Instrument calibration record

1.0 OBJECTIVE

This document defines the standard procedure for measuring water levels in wells. This Standard Operating Procedure (SOP) serves as a supplement to the sampling plan. This procedure describes equipment and field procedures necessary to collect water level measurements. The well locations and frequency of measurement are specified in the plan. This procedure is intended to be used together with the plan and other SOPs.

2.0 EQUIPMENT NEEDED

The equipment necessary to measure water levels includes:

- Solinst Model 101 water level meter or equivalent
- Two 5-gal buckets (with lids) or equivalent for decontamination
- Decontamination brushes
- Alconox soap
- Deionized or distilled water
- Potable water
- Spray bottle
- Field data sheets
- Field notebook
- Appropriate health and safety equipment

3.0 PROCEDURE

Appropriate health and safety equipment, as described in the Health and Safety Plan (HSP) should be worn during well opening, well measurement, and decontamination.

- The water level probe shall be decontaminated prior to use in each monitoring well.
- Observations concerning the well pad, surface or protective casing and other well conditions will be documented in the field notebook.
- The depth of the static water level and the total depth of the well will be measured using an electric water level meter. The measuring point for all the wells shall be the top of stainless steel monitoring well casing. If a reference mark is not found, then all well readings will be referenced to the north rim of the monitoring well riser pipe for standardization.
- The static water level and the total depth of the well shall be measured, recorded

on the water level data sheet, and then immediately rechecked.

- All columns of field data sheets shall be completed, including time of measurement. If measurements are taken over a several-day period, the date of each measurement should be clearly indicated on the form.
- Care shall be taken to verify the readings during each water level measurement period. Any significant changes in water level will be noted by comparing the most recent measurement with past measurements.
- After any measurement is taken, the water level probe shall be decontaminated.
- The water level indicator must be decontaminated before use, between wells, and at the conclusion of measurements.

3.2 DOCUMENTATION

Field data sheets or field notebooks will include date, time, well number, total well depth, water level, static water elevation, and comments. The data sheets or notebook shall be neat and legible, and shall be signed and dated by the person completing the page.

1.0 OBJECTIVE

This document defines the Standard Operating Procedure (SOP) and necessary equipment for collection of groundwater samples in monitoring wells, extraction wells, or piezometers using low-flow techniques. The term "low flow" refers to the velocity that the groundwater is removed from the soil formation immediately adjacent to the well screen.

In this technique, in order to withdraw water from within the well screen and to lessen drawdown, a pump that minimizes disturbance to the groundwater is operated at a low flow rate. The well is only purged within the screened interval until specific parameters have stabilized and as according to the site-specific work plan. Therefore, the groundwater samples collected are representative of the water bearing formation and hydraulically isolated from the water in the casing. The need to purge three well volumes, as required in traditional techniques, is not necessary with low flow purging and sampling. The low flow procedure described in this SOP is not necessarily applicable for every site or for wells screened in materials with very low permeability.

2.0 EQUIPMENT NEEDED

Equipment used during well purging and sampling:

- Well installation forms and boring logs for well being sampled
- Well keys
- Disposable latex or nitrile gloves
- Assorted tools (knife, screwdriver, etc.)
- New synthetic rope
- Pump and required accessories (described in more detail in following section)
- Electronic water level indicator with 0.01-foot increments
- Graduated cylinder
- Temperature meter
- pH meter (with automatic temperature compensation)
- Conductivity meter
- Turbidity meter
- Dissolved oxygen (DO) meter
- Oxidation reduction potential (ORP) meter
- Flow-through cell
- Calibration fluids

- Paper towels or Kimwipes
- Calculator
- Bound field logbook (logbook)
- Waterproof pen and permanent marker
- Plastic buckets
- 55-gallon drums or truck-mounted tank
- Plastic sheeting
- Appropriate decontamination equipment
- Cooler with ice
- Sample containers and labels
- Groundwater sampling form
- Chain-of-Custody form
- Appropriate health and safety equipment (e.g., photoionization detector (PID))

3.0 PROCEDURE

This section provides the step-by-step procedure for collecting groundwater samples in the field. Observations made during groundwater purging and sampling should be recorded in a logbook in accordance with the work plan.

A. Any equipment used in the sampling procedure that could contact groundwater should be properly decontaminated before each use.

B. Equipment should be calibrated based on the manufacturers' instructions. The frequency of calibration should be specified in the site-specific work plan. According to "Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures" (Unites States Environmental Protection Agency (USEPA), 1996), pH calibration should be performed with at least two buffers that bracket the expected range of values. Dissolved oxygen calibration must be corrected for local barometric pressure readings and elevation.

C. Before well purging begins, the following steps should be performed at each well:

- Inspect the well and surrounding site for security, damage, and evidence of tampering. If damage or tampering is evident, contact the project manager for guidance.
- Place clean plastic sheeting around the well (as necessary)

- Measure ambient volatile organic compounds (VOCs) background levels in the immediate vicinity of the well (i.e., using a PID or a flame ionization detector (FID) per the Health and Safety Plan (HASP).
- Remove the well cap and immediately measure VOCs at the rim of the well and record the readings in the logbook or on the groundwater sampling form. Give the water in the well adequate time to reach equilibrium.

D. After the well has reached equilibrium, the groundwater elevation should be measured to the nearest 1/100-foot. The total well depth and screened interval should be obtained from the well logs. Measuring the total depth prior to sampling should be avoided to prevent resuspension of settled solids in the well casings and to minimize the necessary purge time for turbidity equilibration. The total depth of the well should be confirmed after sampling has been completed.

E. Following measurement of the static groundwater elevation, the appropriate equipment will be slowly and carefully placed in the well. If the wells have light or dense non-aqueous-phase liquids (LNAPLs or DNAPLs) care should be taken to place sampling equipment below or above the NAPL.

Selection of the proper pump is important for low-flow sampling activities. USEPA guidance (1996) notes that dedicated sampling devices capable of purging and sampling are preferred over any other type of device. In addition, the pump must be capable of flow rates between 0.1 and 1.0 liter per minute. A variety of portable sampling devices are available, such as bladder pumps, electrical submersible pumps, gas-driven pumps, inertial lift foot-valve samplers (e.g. check-ball systems), and bailers (a list of pump manufacturers and suppliers is included on pg. 7).

When placing the equipment in the well, the pump intake should be set near the middle or slightly above the middle of the screened interval. If the screen length allows, the pump intake should be at least two feet from the bottom of the screen. Placing the pump intake near the top of the water column can cause stagnant water from the casing to be purged, but placing the pump intake near to the bottom of the well can cause mobilization and entrainment of settled solids from the bottom of the well.

F. Tubing should be connected from the pump to a flow-through cell. Then, calculate the volume of water to fill the flow-through cell. According to American Society for Testing and Materials (ASTM) Standard D 6771 (2002), the frequency of measurements should be equal

to the time required to completely evacuate one volume of the cell. This ensures that independent measurements are made.

G. The pump should be started at a low flow rate, approximately 100 mL/min or the lowest flow rate possible.

H. Water level measurements should continue every two minutes until the measurements indicate that significant drawdown is not occurring. According to ASTM standards (2002), allowable drawdown should never exceed the distance between the top of the well screen and the pump intake. Including a safety factor, also provided by ASTM, drawdown should actually not exceed 25% of this distance. This ensures that water stored in the casing is not purged or sampled. For example, for a 4-foot screen, the pump should be placed at the midpoint of the screen (two feet from the top of the screen to the pump intake). With a safety factor of 25%, this would require drawdown not to exceed six inches.

Once it has been established that significant drawdown is not occurring, the flow rate may be increased to </= 1 L/min (ASTM, 2002) or, if the flow rate remains the same, water level measurements need only to be taken periodically. However, when the flow rate is increased, water level measurements must continue every two minutes.

If drawdown surpasses 25% of the distance from the pump intake to the top of the screen even while pumping is occurring at the lowest flow rate possible, samplers should refer to the project specific criteria as found in the appropriate FSP or work plan.

I. Parameters should be documented on the groundwater sampling form and in the logbook. The time between parameter measurements is calculated as follows:

$$T = \frac{V}{Q}$$, where

T = time between measurements (minutes)
V = volume of the flow-through cell (liters)
Q = purge flow rate (liters per minute)

J. Sampling should occur as stated in the FSP or work plan. However, in most cases, purging will continue until specific parameters have stabilized over three consecutive flow-through cell volumes. **Table 1** provides guidelines that may be used for parameter stabilization as specified by ASTM Guidance on Low-Flow Purging and Sampling and Minimum-Purge Sampling. These guidelines are to be used in combination with professional judgment.

Table 1. Stabilization Guidelines for Low-Flow Sampling

Parameter	Stabilization Guidelines
	ASTM
DO	+/- 10% or +/-0.2 mg/L, whichever is greatest
ORP	+/- 20 mV
PH	+/- 0.2 units
Conductivity	+/- 3%
Temperature	Not Specified
Turbidity	Not Specified

K. After the relevant parameters have stabilized, the flow-through cell should be disconnected or bypassed for sampling. If, after a considerable number of readings have been taken, parameters have not stabilized, samplers should refer to the work plan or possibly use alternative sampling methods.

L. The flow rate should be adjusted to less than 0.5 L/min for sampling to minimize aeration during the sampling of volatiles.

M. A new pair of disposable latex or nitrile gloves should be put on immediately before sampling.

N. The constituents should be sampled for in the order given below:

- VOCs – Vials should be filled completely so that the water forms a convex meniscus then capped so that no air space exists in the vial. Turn the vial over and tap it to check for bubbles. If air bubbles are observed in the sample vial, remove the lid and attempt to fill the vial two more times, (being careful not to dump out any groundwater currently in the vial). If air bubbles are present twice more, discard the sample vial and repeat the procedure with a new vial. If, after three attempts, air bubbles are still in the vial, make a note of this and place the vial in the cooler.
- Gas sensitive parameters (e.g., ferrous iron, methane, alkalinity)
- Semivolatile organic compounds, pesticides, polychlorinated biphenyls, and herbicides
- Petroleum hydrocarbons
- Metals (unfiltered)
- Explosives
- Any filtered analytes (use in-line filters if possible)

O. Place all samples on ice inside a cooler immediately.

P. Each sample should be identified with the Sample ID, location, analysis number, preservatives, date and time of sampling event, and sampler.

Q. The sample time and constituents to be analyzed for should be recorded in the logbook and on the groundwater sampling form.

R. Chain-of-custody procedures should be started.

S. Sample equipment should be decontaminated.

T. The well sampling order should be dependent on expected levels of contamination in each well, if known, and should be determined prior to sampling. Sampling should progress from the least contaminated to the most contaminated well. Quality assurance/quality control (QA/QC) samples should be collected during groundwater sampling as required in the work plan and/or QAPP.

LIST OF SUPPLIERS WHO PROVIDE PUMPS SUITABLE FOR LOW-FLOW SAMPLING:

Field Environmental. 1-800-3930-4009. www.fieldenvironmental.com. Pumps: peristaltic, QED bladder pumps, Fultz rotor pump, control boxes, compressors, etc.

QED. 1-800-624-2026. www.micropurge.com. Pumps: bladder pumps, flow cell, compressors, etc.

Fultz Pumps. 1-717-248-2300. www.fultzpumps.com.

REFERENCES

ASTM 2002, Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations, ASTM D6771-02, American Society for Testing and Materials. West Conshohocken, PA.

Nielsen, David and Nielsen, Gillian. Technical Guidance on Low-Flow Purging and Sampling and Minimum-Purge Sampling. Second Edition. NEFS-TG001-02. April 2002.

USEPA. 1996. Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures. EPA/540/S-95/504. OSWER, April, 1996.

1.0 OBJECTIVE

This document defines the standard protocols for sample handling, documentation, tracking, and control and custody of environmental samples. This SOP serves as a supplement to the work plan.

2.0 EQUIPMENT NEEDED

The following equipment will be needed for sample control and custody procedures:

- Waterproof coolers (hard plastic or metal)
- Custody Seals
- Field forms such as a Chain of Custody (COC) or sample collection sheet
- Field Notebook
- Ice
- Sample Log-in Book
- Clear Tape
- Duct Tape
- Zip-Loc Bags
- Waterproof pens
- Permanent Markers.

3.0 PROCEDURES FOR SAMPLE IDENTIFICATION, HANDLING, AND DOCUMENTATION

Sample Identification

Samples collected during site activities shall have discrete sample identification numbers. These numbers are necessary to identify and track each of the many samples collected for analysis during the life of this project. In addition, the sample identification numbers will be used in the data base to identify and retrieve the analytical results received from the laboratory.

Each sample is identified by a unique code which indicates the site identification number, sample location number, sample matrix identifier, and sample depth.

The sample identification system for groundwater sampling will involve the following

nomenclature "AAA-BB" where:

"AAA" will denote the monitoring well

- GM8- Monitoring Well Location

"BB" will denote QA/QC sample

- EB- equipment blank
- AD- analytical duplicate
- MS or MD – Matrix Spike or Matrix Duplicate
- TB- Trip Blank

The sample identification system for soil will involve the following nomenclature "S-AA-BB-C-D-EEE" where:

"AA" will denote

- ## - Sample Area

"BB" will denote

- ##- Sample Number

"C" will denote

- #- Initial Sample Depth

"D" will denote

- #- Final Sample Depth

"EEE" will denote QA/QC sample

- DUP- analytical duplicate
- MS or MD – Matrix Spike or Matrix Duplicate

For example, S-04-18-1-2 indicates the soil sample was obtained from sample area 4, the North Production Area, for boring number 18, from 1 to 2 ft bgs.

Sample Labeling

Sample labels will be filled out as completely as possible by a designated member of the sampling team prior to beginning field sampling activities each day. The date, time, sampler's

signature, and the last field of the sample identification number should not be completed until the time of sample collection. All sample labels shall be filled out using waterproof ink. At a minimum, each label shall contain the following information:

- Project name and number,
- Sample number identification,
- Initials of sampler,
- Sampling location (if not already encoded in the sample number),
- Required analysis,
- Date and time of sample collection,
- Space for laboratory sample number, and
- Preservative used, if applicable.

Sample Handling and Shipping

The sample containers will be placed in reclosable Ziploc plastic storage bags and wrapped in protective packing material (bubble wrap). Samples will then be placed right side up in a cooler with ice (double bagged using plastic bags). Once the samples are collected, they must remain in the custody of the sampler or another worker from the site. The samples can also remain unattended in a locked vehicle so tampering with the samples will not be possible. Right before shipment, a custody seal should be placed over the opening of the cooler and then the cooler should be taped all the way around with clear packing tape to prevent tampering with the samples. Samples will be hand delivered or shipped by overnight express carrier for delivery to the analytical laboratory. All samples must be shipped for laboratory receipt and analyses within specific holding times. This may require daily shipment of samples with short holding times. A chain-of-custody (COC) form will accompany each cooler. The temperature of all coolers will be measured upon receipt at the laboratory. A temperature blank will be included in each cooler for temperature measurement purposes.

Field Notes

Documentation of observations and data acquired in the field will provide information on the acquisition of samples and also provide a permanent record of field activities. The observations and data will be recorded using pens with permanent waterproof ink in a permanently bound weatherproof field log book containing consecutively numbered pages.

URS personnel will keep a bound field notebook while performing sampling and oversight activities on-site. Forms that will be used include: chain-of-custody, boring log, and soil sampling data sheets and field logbook. The field logbooks will record the following:

- Changes to the plan;
- Personnel conducting the site activities, their arrival and departure times and their destination at the site;
- Location where the work was performed;
- Date, time, weather conditions, equipment, and personnel on site;
- Daily information such as:
  - Work accomplished and the current site status,
  - Equipment calibrations, repairs and results, and
  - Site work zones;
- Specific work activities conducted such as
  - Work zone and headspace readings; and
- Incidents and unusual activities that occur on the site such as, but not limited to, accidents, breaches of security, injuries, equipment failures, or weather related problems.

The following sampling-related information will be recorded in the field logbook by the field sampling team:

- Sample number,
- Project identification,
- Sampling location,
- Required analysis,
- Date and time of sample collection,
- Type and matrix of sample,
- Sampling technique,
- Preservative used, if applicable,
- Sampling conditions,
- Observations,
- Initials of the sampler, and
- Samples collected.

Each page in the field books will be signed by the person making the entry at the end of the day, as well as on the bottom of each page. Anyone making entries in another person's field book will sign and date those entries. Any entry which is to be deleted will have a single cross out which is signed and dated. Sufficient information should be recorded to allow the sampling event to be reconstructed without relying on the sampler's memory. Photographic records will be developed through the use of digital photographs, showing pre-sampling and post-sampling conditions at each site.

Procedures to evaluate field data for this project primarily include checking for transcription errors on the part of field crew members and review of field notebooks. This task will be the responsibility of the URS Field Leader, who will otherwise not participate in making any of the field measurements or in adding notes, data, or other information to the notebook.

Sample Chain-of-Custody

During field sampling activities, traceability of the samples must be maintained from the time the samples are collected until the laboratory data is issued. Initial information concerning collection of the samples will be recorded in the field log book as described above. Information on the custody, transfer, handling, and shipping of samples will be recorded on a COC form.

The sampler will be responsible for initiating and filling out the COC form. The COC will be signed by the sampler or the field person responsible for sample handling when the sampler relinquishes the samples to anyone else. One COC form will be completed for each cooler of samples collected daily and if samples are not hand delivered, the COC will be placed in a Zip-Loc bag and shipped inside the cooler. COC forms will be used to document the transport and receipt of samples from the field to the lab. Information required on a COC includes the following:

- Sampler signature/date/time/affiliation,
- Project number,
- Date and time of collection,
- Sample identification number,
- Sample type and matrix,
- Sample description/location,
- Analyses requested/comments,

- The total number of containers being sent to the lab and number of containers for each sample,
- The appropriate preservative used,
- If any samples are to be placed on hold at the laboratory, this should be clearly indicated on the COC in the comments section,
- Signature of person(s) relinquishing custody, dates, and times,
- Signature of person(s) accepting custody, dates, and times,
- Method of shipment, and
- Shipping air bill number.

The person responsible for delivery of the samples to the laboratory will sign the COC form, retain the last copy of the three-part COC form, document the method of shipment, and send the original and the second copy of the COC form with the samples. Upon receipt at the laboratory, the person receiving the samples will sign the COC form and return the second copy to the Project Manager. Copies of the COC forms documenting custody changes and all custody documentation will be received and kept in the central files. The original COC forms will remain with the samples until final disposition of the samples by the laboratory. The analytical laboratory will dispose of the samples in an appropriate manner 60 to 90 days after data reporting. After sample disposal, a copy of the original COC will be sent to the Project Manager by the analytical laboratory to be incorporated into the central files.

CHAIN OF CUSTODY RECORD

SHEET ___ of ___

URS CORPORATION

1001 HIGHLAND PLAZA DRIVE WEST, SUITE 300
ST. LOUIS, MISSOURI 63110
314-429-0100

PROJECT NO:	PROJECT NAME:	NO. OF CONTAINERS	CONTAINER DESCRIPTION / ANALYSES REQUESTED	REMARKS
SAMPLER'S: (Signature)				
DATE / TIME	SAMPLE I.D. NUMBER			

RELINQUISHED BY: (Signature)	DATE / TIME	RECEIVED BY: (Signature)	DATE / TIME
RELINQUISHED BY: (Signature)	DATE / TIME	RECEIVED AT LAB BY: (Signature)	DATE / TIME
METHOD OF SHIPMENT:		AIRBILL NO:	

1.0 OBJECTIVE

This document defines the Standard Operating Procedure (SOP) for sample handling, documentation, and tracking. This SOP serves as a supplement to the plan.

2.0 EQUIPMENT NEEDED

The following equipment will be required for this SOP:

- Waterproof coolers (hard plastic or metal)
- Custody Seals
- Field forms such as COC or sample collection sheet
- Field Notebook
- Ice
- Bubble Wrap
- Clear Tape
- Duct Tape
- Zip Loc Bags
- Sample Containers
- Waterproof Pen
- Permanent Marker.

3.0 SAMPLE CONTAINERS

Certified commercially clean sample containers will be obtained from the contract analytical laboratory. The lab will indicate the type of sample to be collected in each bottle type. The work plan lists the appropriate sample containers for the specific analyses require for each project.

4.0 SAMPLE PRESERVATION

Samples will be preserved at the time of the sample collection. Chemical preservatives, if necessary, will be added to the sample containers either by the laboratory prior to shipment to the field, or in the field by sampling personnel.

After sample collection, each container will be labeled and stored on ice at 4°C in an insulated cooler until packed for shipment until packed for shipment to the laboratory. The ice will be double bagged in Zip Loc storage bags. Freezing samples will not be permitted. Any breakable

sample bottles need to be wrapped in protective packing material (bubble wrap) to prevent breakage during shipping.

5.0 SAMPLE HOLD TIMES

Samples will be hand delivered or shipped by overnight express carrier for delivery to the analytical laboratory. All samples must be shipped for laboratory receipt and analyses within specific holding times. This may require daily shipment of samples with short holding times. The hold time varies for each type of analysis. It will be necessary to check with the lab to verify the hold times to determine how frequently samples need to be sent to the lab.

Documentation of observations and data acquired in the field will provide information on the acquisition of samples and also provide a permanent record of field activities. The observations and data will be recorded using pens with permanent waterproof ink in a permanently bound weatherproof field log book containing consecutively numbered pages.

1.0 OBJECTIVE

This document defines the Standard Operating Procedure (SOP) for decontamination of equipment used in environmental sites.

2.0 EQUIPMENT NEEDED

The following equipment will be needed for decontamination procedures:

- Brushes
- Wash Tubs
- Buckets
- Scrapers, flat bladed
- Hot water – high pressure washer
- Paper towels
- Alconox detergent (or equivalent)
- Potable tap water
- Laboratory-grade deionized or distilled water
- Garden-type water sprayers

2.0 SAMPLING EQUIPMENT DECONTAMINATION PROCEDURES

Sampling equipment will be decontaminated at the sampling location under the following procedures:

- Personnel will wear the proper PPE to reduce the potential for exposure as required by the HASP.
- Partially fill two buckets with potable tap water, and add Alconox detergent to one of the buckets
- Use brushes to wash the sampling equipment (i.e. stainless steel bowls, stainless steel spoons, sampling utility knife, etc)
- Rinse sampling equipment in bucket containing potable tap water
- Rinse clean equipment with water sprayers containing distilled water (or equivalent)
- Place decontaminated equipment in clean area and allow to air dry.

3.0 DRILLING AND HEAVY EQUIPMENT DECONTAMINATION PROCEDURES

Drilling rigs will be decontaminated at a decontamination station located near a staging area. The decontamination station may be a temporary structure, or mobile trailer, capable of collecting all decontamination fluids. The following steps will be used to decontaminate drilling and heavy equipment.

- Personnel will suit up in proper PPE to reduce the potential for exposure as required by the HASP.
- Equipment showing gross impacted soil materials will be scrapped with a flat-bladed scrapper, and material containerized.
- Equipment that cannot be damaged by water, such as a drill rig, augers, drill bits, sampling equipment, shovels, etc, will be washed with a hot water, high-pressure sprayer, the rinsed with potable water.
- Following decontamination, drilling equipment will be placed on the clean drill rig and moved to the next sampling location. If equipment is not immediately used, it should be stored in a clean designated area.

Equipment rinsante samples of the decontaminated sampling equipment may be collected to verify the effectiveness of the decontamination procedures.

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) outlines the procedures for soil sample logging. The purpose of soil sample logging is to record all field notes in a detailed and organized fashion.

2.0 PROCEDURES

At the outset of sample logging, the on-site geologist will record field notes with waterproof ink in a bound field notebook. At a minimum, the daily field notes will include:

- Project name and number
- Date and time
- Weather conditions
- Sampler's name
- Project objective(s).

Throughout the sampling round, the following items will be recorded as appropriate:

- Sample location(s)
- Sample identifications
- Limiting field conditions
- Problems encountered.

Unconsolidated soil samples will be described as follows:

- Descriptive information:
  - Color name (Munsell Color Chart) of the logged interval or sample
  - Color notation including chroma, hue, value, and qualifiers

1. Mottling with abbreviations, descriptors, and criteria for descriptions of mottles as identified below

Descriptors for Mottling

Abundance	Size	Contract
f: few (<2%)	fine (<5 mm)	faint
c: common (2%-20%)	medium (5-15 mm)	distinct
m: many (>20%)	coarse (>15 mm)	prominent

2. Degree of saturation (dry, damp, moist, wet, saturated, or combinations); note depth to groundwater table, if observed

3. Degree of density. Count the blows of each 12-inch increment of the sampler (ASTM-1586-84). Use the values and the density table presented below to determine the degree of density.

Degree of Density

Cohesive Clays		Non-cohesive Granular Soils	
<2	very soft	0-4	very loose
2-4	soft	>4-10	loose
>4-8	medium stiff	>10-30	medium dense
>8-15	stiff	>30-50	dense
>15-30	very stiff	>50	very dense
>30	hard		

4. Soil description according to ASTM's Unified Soil Classification System (USC) and by soil structure:

   – ASTM Unified Soil Classification: The Grade Limits and Grade Standards table presented below provides the grade limits and grade names used by engineers according to ASTM standards D422-63 and D643-78.

Grade Limits and Grade Standards

Grade Limits		Grade Names	
mm	inch	US standard sieve series	
			boulders
305	12.0		
			cobbles
76.2	3.0	3.0 inch	
			gravel
4.75	0.19	No. 4	
2.00	0.08	No. 10	
			medium sand
0.425		No. 40	
0.074		No. 200	
			silt
0.005			
			clay size

Source: AGI data sheet 29.2

   – Course-grained soils include clean gravels and sands and silty or clayey gravels and sands with more than 50% retained on the No. 200 sieve. A table of USC symbols and names for coarse-grained soils is presented below.

USCS Symbols and Names for Coarse-grained Soils

USCS Symbol	Typical Names
GW	Well graded gravels, gravel-sand mixtures, little or no fines
GP	Poorly graded gravels, gravel-sand mixtures, little or no fines
GM	Silty gravels, gravel-sand-silt mixtures
GC	Clayey gravels, gravel-sand-clay mixtures
SW	Well graded sands, gravelly sands, little or no fines
SP	Poorly graded sands, gravelly sands, little or no fines
SM	Silty sand, sand-silt mixtures
SC	Clayey sands, sand-clay mixtures

- Fine-grained soils include inorganic and organic silts and clays; gravelly, sandy, or silty clays; and clayey silts with more than 50% passing the No. 200 sieve. A table of USC symbols and names for fine-grained soils is presented here.

USCS Symbols and Names for Fine-grained Soils

USCS Symbol	Typical Names
ML	Inorganic silts and very fine sands, rock flour, silty, or clayey fine sands, or clayey silts with slight plasticity
CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays
OL	Organic silts and organic silty clays of low plasticity
MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts
CH	Inorganic clays or high plasticity (residual clays), fat clays
OH	Organic clays of medium to high plasticity, organic silts
Pt	Peat and other highly organic soils

- A table of soil descriptors is presented below.

Soil Descriptors

Calcareous:	containing appreciable quantities of calcium carbonate
Fissured:	containing shrinkage cracks, often filled with fine sand or silt, usually more less vertical
Interbedded:	containing alternating layers of different soil types
Intermixed:	containing appreciable, random, and disoriented quantities of varying color, texture, or constituency
Laminated:	containing thin layers of varying color, texture, or constituency
Layer:	thickness greater than 3 inches

Soil Descriptors

Mottled:	containing appreciable random speckles or pockets of varying color, texture, or constituency
Parting:	paper thin
Poorly graded (well sorted):	primarily one grain size, or having a range of sizes with some intermediate size missing
Slickensided:	having inclined planes of weakness that are slick and glossy in appearance and often result in lower unconfined compression cohesion
Split graded:	containing two predominant grain sizes with intermediate sizes missing
Varved:	sanded or layered with silt or very fine sand (cyclic sedimentary couplet)
Well graded (poorly sorted):	containing wide range of grain sizes and substantial amounts of all intermediate particle sizes
Modifiers:	Predominant type - 50% to 100% Modifying type - 12% to 50% With - 5% to 12% Trace - 1% to 5%

5. Degree of plasticity. The following table presents the terms used to denote the various degrees of plasticity of soil that passes the No. 200 sieve.

Degrees of Plasticity

Descriptive Term	Degree of Plasticity	Plasticity Index Range
SILT	none	non-plastic
Clayey SILT	slight	1-5
SILT & CLAY	low	5-10
CLAY & SILT	medium	10-20
Silty CLAY	high	20-40
CLAY	very high	over 40

6. Drilling information:

   - Drill rig manufacturer, model, and driller (if applicable)
   - Geologist or geotechnical engineer
   - Project name, sample point identification, and location
   - Date samples obtained (and times if required)
   - Type of sampler (e.g., split spoon, Shelby, California), measurements or method of advancing boring or equipment, method of driving sampler, and weight of hammer
   - Drill fluids (if applicable)

- Ground surface or grade elevation (if known)
- Depth penetrated and blow counts/6-inch interval of penetration for ASTM 1586-84 and sample number (if applicable)
- Closed hole intervals and advancement (if applicable)
- Recovery
- Strata changes and changes within samples
- Sampling tool behavior
- Drill string behavior
- Use(s) of borehole
- Disposition(s) of residual soil or cuttings
- Signature or sampling of log (as required)

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) describes the procedure to obtain representative subsurface soil samples for geologic logging and physical and chemical laboratory testing.

2.0 EQUIPMENT NEEDED

The following equipment is typically required:

- Hydraulic percussion hammer Geoprobe
- 1 inch diameter by 3 foot length steel probe rods
- Barrel sampler - 2 1/4 in diameter by 4 ft length
- Acetate liners
- Disposable sample retainers
- Photoionization detector (PID, OVM)
- Surveyor's stakes
- Stainless steel pans, knives and plastic Zip-loc bags
- Sample containers
- Decontamination equipment.

3.0 PROCEDURE

The general procedure for using the Geoprobe equipment for sampling is as follows:

1. Locate boring using facility drawings to check utilities
2. Log boring location on site base map
3. Hydraulically push or drive 1 in. diameter probe rods with barrel sampler attached to the first sample depth
4. Remove barrel sampler and retrieve acetate liner. Visually log and classify the soil, select specimen for physical and/or chemical testing. Record information on field data sheets
5. Decontaminate barrel sampler and install new acetate liner
6. Measure VOC concentrations with PID at top of probe hole prior to sampling the next depth interval (if VOCs are a concern)
7. Insert barrel sampler in exiting probe hole and push or drive sampler to the next sample depth, repeat sampling procedure
8. Repeat Geoprobe sampling until the target depth is reached
9. Record total depth
10. Retrieve probe rods

11. Backfill probe hole with bentonite
12. Place survey stake at boring location
13. Record data collected on boring log and log book
14. Decontaminate equipment.

4.0 DECONTAMINATION

Refer to the HSP for personnel decontamination procedures; refer to the equipment decontamination procedures for equipment decontamination procedures.

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) describes the methods to be used for the calibration and use of the Photoionization Detector (PID) for field headspace analysis and health and safety monitoring.

2.0 PURPOSE

The purpose of this procedure is to develop and maintain good quality control in field operations and to create uniformity between field personnel involved with PID use.

3.0 EQUIPMENT NEEDED

PID (probes with 11.7 eV lamp or equivalent) or suitable for site conditions, log book, user's manual, calibration gas.

4.0 PROCEDURE

Calibration:

1. Prior to calibration, check the function switch on the control panel to make sure it is in the "OFF" position. The probe nozzle is stored inside the instrument cover. Remove cover plate by pulling up on the pins that fasten the cover plate.
2. Remove the nozzle from the cover. Assemble probe by screwing nozzle into casing.
3. Attach probe cable to instrument box inserting 12 pin interface connector of the probe cable into the connector on the instrument panel. Match the alignment keys and insert connector. Turn connector in clockwise direction until a distinct snap and lock is felt.
4. Turn the function switch to the Battery Check position. When the battery is charged, the needle should read within or above the green battery arc on the scale plate. If the needle is below the green arc or the red LED light comes on, the instrument should be recharged prior to making any measurements.
5. Turn the function switch to the "ON" position. In this position, the UV light source should be on. To verify, glance at the end of the probe for a purple glow. Do Not Look Directly at the Lamp Itself. If the lamp does not come on refer to the Instruction Manual.
6. To zero the instrument, turn the function switch to the standby position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counter clockwise rotation yields a downscale deflection. (Note: No zero gas is needed since this is an electronic zero adjustment.) If the span adjustment is changed during instrument calibration, the zero should be rechecked and adjusted. If necessary, wait 15 to 20 seconds to ensure that the zero reading is stable. Readjust as necessary.

Instrument Daily Calibration:

1. Insert one end of T-tube into probe. Insert second end of probe into calibration gas in the 20-200 ppm range. The third end of probe should have the rotometer (bubble meter) attached.
2. Set the function switch in the 0-200 ppm range. Crack the valve on the pressured calibration gas container until a slight flow is indicated on the rotameter. The instrument will draw in the volume required for detection with the rotameter indicating excess flow.
3. Adjust the span potentiometer so that the instrument is reading the exact value of the calibration gas. (Calibration gas value is labeled on the cylinder.)
4. Turn instrument switch to the standby position and check the electronic zero. Reset zero potentiometer as necessary following step 6 above.
5. Record all original and readjusted settings in log book.
6. Set the function switch to 0-20 ppm. Remove the mid-range (20-200 ppm) calibration gas cylinder and attach the low-range (0-20 ppm) calibration gas cylinder as described above.
7. Do not adjust the span potentiometer. The observed reading should be ± 3 ppm of the concentration specified for the low-range calibration gas. If this is not the case, recalibrate the mid-range scale repeating Steps 1 through 6 above. If the low-range reading consistently falls outside the recommended tolerance range, the probe light source window likely needs cleaning. Clean window according to instruction manual. When the observed reading is within the required tolerances, the instrument is fully calibrated.

Instrument Calibration Check:

1. Exit the exclusion zone and turn meter to "ON" position. Check that the meter is reading a value of zero.
2. Insert one end of T-tube into probe and other end into calibration gas. The third end of the T-tube should be attached to a flow meter.
3. Crack the valve on the calibration gas and read the value shown by the instrument. Record the value and calibration gas concentration on a field-data sheet.
4. If the value shown by the instrument is greater than $\pm 20\%$ of the calibration gas concentration, take meter outside of exclusion zone and recalibrate as outlined above.

Sample Measurement:

1. Place function switch in 0-20 ppm range for field monitoring. This will allow for most sensitive, quick response in detecting airborne contaminants.

2. Before entering a contaminated area, determine background concentration. This concentration should be used as a reference to readings made in the contaminated area. Under no circumstance should one attempt to adjust the zero or span adjustments while the instrument is being operated in the field.
3. Take measurements in contaminated area, recording readings and locations. Should readings exceed the 0-20 scale, switch the function switch to the 0-200 or 0-2,000 range as appropriate to receive a direct reading. Return the instrument switch to the 0-20 range when readings are reduced to that level. Record measurements on field-data sheet.
   Note: The instrument will not function properly in high humidity or when the window to the light housing is dirty. If the instrument response is erratic or lower than expected, recalibrate or obtain a different meter and calibrate as outlined above.
4. When finished, reverse Steps 1 through 6 in Instrument Setup section to shut down the instrument.

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) describes the methods to be used in measuring organic vapors emitted from soils collected with a mechanical device or hand augering device. Results will be used as a field screening for volatile organic vapors.

2.0 PURPOSE

The purpose of this procedure is to maintain uniformity between field personnel performing the measurements and to provide representativeness of readings obtained.

3.0 EQUIPMENT NEEDED

Personal protective equipment, PID, wide-mouth sample jars and aluminum foil or polyethylene bags (Ziploc type), rubber bands, field data forms.

4.0 PROCEDURES

1. Samples are collected and placed in wide-mouth sample jars or polyethylene bags (zip-loc type) so that the jars or bags are approximately half full. The jars or bags are labeled to document sample location and depth, time, date, and field personnel collecting the sample.
2. The glass jar is capped with aluminum foil, a rubber band, and the lid, if it will fit or the bag is zipped shut.
3. The air-tight sample container is then allowed to warm for at least 10-15 minutes to allow the liberation of soil gases into the headspace.
4. Calibrate and prepare PID for use.
5. Puncture the aluminum foil or polyethylene bag with the calibrated monitor probe and allow headspace gases to be drawn through the PID unit.
6. Record the highest response obtained on an appropriate sampling log.
7. Remove the punctured foil and seal jar with the proper lid.
8. Allow instrument to return to zero and repeat procedure for next sample.

1.0 OBJECTIVE

Collection of soil samples for VOC analysis that will minimize the loss of contaminants due to volatilization and biodegradation

2.0 EQUIPMENT

The following equipment is required for each sample point.

- One Terra Core™ (or equivalent)
- Two (40-ml VOA vials with sodium bisulfate preservative)
- One (40-ml VOA vial with methanol);
- One 2 or 4-oz glass jar for screening and/or high level analysis, and dry weight conversions (or as specified by laboratory)
- Paper towels
- Indelible pen
- Clear Tape and Labels.

3.0 PROCEDURE

1. The following general procedures are followed for collection of soil samples with the Terra Core™ sampler
2. Remove plunger from package and seat in the handle of the sampler
3. Push the plunger and sampler straight down into a freshly exposed surface of soil until the sample chamber is full (approximately 5 grams of soil)
4. Slowly remove plunger and sampler and inspect bottom of sampler. If sampler is not full, repeat step 3
5. Remove excess soil from the sampler mouth, the soil plug should be flush with the mouth of the sampler
6. Rotate the plunger that was seated in the handle top 90 degrees until it is aligned with the slots in the body
7. Lower the sampler to the top of one of the 40-mil preserved VOA vials and extrude the sample by pushing the plunger down
8. Quickly place the lid back on the 40-ml VOA vial, making sure vial threads are clear of soil and debris
9. Repeat procedures 2 through 8 to fill the other two preserved vials
10. Collect additional soil and place in 2 or 4 oz glass jar to be used for dry weight conversion.
11. Place sample labels on sample containers
12. Secure label with clear tape and place in cooler, keep sample at 4 degrees Celsius

VOLATILE COMPOUNDS BY GC/MS (EPA 8260B)

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Approved by: _____ 3 April 2001
Date

Title: Technical Manager, QA

STL X Savannah ___ Tallahassee ___ Mobile ___ Tampa West

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures that can be used to determine the concentration of volatile organic compounds (VOC) in water, wastewater, soils/sediments, wastes, oils, sludges, and solids. The attached quantitation report lists the target compounds, an example of the retention time order of each target compound, the quantitation and confirmation ions of the target compounds, and internal standard assignments.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision criteria for each target compound are listed in Section 5 of the current revisions of the STL Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan.*

2.0 SUMMARY OF METHOD

2.1 Volatile organic compounds (VOC) are purged from the sample matrix with helium. The VOC are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOC are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOC, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.2 Aqueous samples may be purged at ambient conditions (recommended) or at 40C (optional). Five to twenty-five milliliter aliquots of the sample may be purged. The calibration standards and the associated QC must be analyzed under the same conditions and volume.

2.3 Low-level (nominally <1mg/kg) soil samples are purged at 40C in a purge and trap instrument designed to add water and internal standards to the vial containing the sample without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards are purged under the same conditions.

2.4 High level soils (nominally >1mg/kg) and waste samples are extracted with methanol-1mL of methanol per gram of sample. An aliquot of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at ambient temperature using the same instrument conditions and calibration used for aqueous samples.

2.5 This method is based on the guidance in SW-846 Methods 8260B and 5035.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedure that you do not understand or that will put yourself or others in a potentially hazardous situation.

3.2 Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, eye protection, or other equipment should be used. Standards and highly contaminated samples should be handled in a hood.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to safely handle these materials.

3.4 The exit vent of the split injector must have a carbon trap in-line to collect the volatile compounds that are vented during the injection of the sample. The traps should be changed a minimum of every three months and disposed of in accordance with STL-SL SOP CA70: *Waste Management.*

4.0 INTERFERENCES

4.1 VOCs commonly used in the laboratory are potential sources of contamination. Methylene chloride, acetone, Freon-113, MEK, hexane, toluene, and isopropanol are used in the laboratory and tend to present the most problems.

4.2 The volatiles lab must be kept as free from contamination as possible. Highly contaminated samples must be segregated from routine samples. Contact with sections of the laboratory where solvents are used should be minimized. Refrigerator blanks should be prepared, stored, and analyzed to evaluate the sample storage areas for possible contamination. Guidance is provided in STL-SL SOP AN70: *Segregation of Low and High Concentration Volatile and Semivolatile Samples.*

4.3 Matrix interferences may be overcome by the use of the secondary ions for quantitation. An example of this is the use of mass 82 for quantitation with chlorobenzene-d5 internal standard when a potential co-eluter, 1,1,1,2-terachloroethane, is a target compound. One of the mass fragments of 1,1,1,2-tetrachloroethane is mass 117, which is the recommended quantitation ion for chlorobenzene-d5. The use of the secondary ions should be used for quantitation in such cases when the lab can clearly demonstrate matrix problems. Mass 58 is recommended for quantitation of acetone due to the elution of a hydrocarbon at the same retention time.

4.4 The analysis of highly contaminated samples (>1mg/L or >1mg/kg) can affect succeeding analyses. Carry-over can occur when low concentration samples are analyzed after high concentration samples. Trap replacement and purging of the entire purging system may be necessary when carry-over is suspected. Reagent blanks must be analyzed when carryover is suspected to demonstrate that the system is free from contamination.

4.5 The Teflon seals of the purge and trap device can absorb and outgas many of the compounds that are included in this method. These Teflon fittings should be periodically checked for integrity. If contamination of the fittings is suspected, the fittings may be heated at 105 C for one hour or replaced.

5.0 SAMPLE COLLECTION, HANDLING, AND PRESERVATION

5.1 Liquid samples are collected with no headspace in 40mL vials equipped with Teflon-lined caps. The samples are acidified at the time of collection with about 0.10mL of concentrated HCl per 40mL of sample. The acid prevents the biological degradation of the aromatic compounds and prevents the dehydrohalogenation of some of the chlorinated alkanes. The sample must be iced at the time of collection and refrigerated at 4C (less than 6C with no frozen samples) in the lab until analysis.

Check each sample vial at the time of receipt for the presence of "bubbles". If the bubbles are less than 3mm in diameter, the vial is acceptable. If the bubble is greater than 3mm, use another vial. Notify the department supervisor or project manager if there are no acceptable vials for analysis.

A "sacrificial" vial or the vial used for screening analysis is used to check the sample pH. If the sample pH is greater than 2, notify the department supervisor or project manager. If directed by supervisor or project manager, hydrochloric acid may be added through the septum to bring the pH <2. Do not add more than 400uL (0.40mL) of 1:1 HCl to a VOC vial. If pH cannot be adjusted to <=2 without destroying the integrity of the sample, the sample must be analyzed within 7 days of collection.

The holding time for samples preserved with HCl is 14 days for all target compounds. The holding time for un-preserved samples is 7 days.

5.2 Soils: Soils are routinely collected in duplicate in Encore samplers. A "bulk" sample is also routinely collected in a 125-mL jar fitted with a Teflon-lined cap. The bulk sample can be used for the methanol extraction if the concentration of the sample collected in the Encore exceeds the working range of the analytical system.

Soils collected in Encore samplers must be analyzed within 48 hours of collection or must be transferred within 48 hours of collection to sealed vials containing sodium bisulfate solution or methanol. If the sample contains high levels of carbonates, the sample is preserved with water and frozen until the time of analysis. The procedure for preparing soil samples is given in Section 9.2.

The hold time of the preserved sample is 14 days from the date of collection. The hold time for frozen samples is 14 days from the date of collection.

5.3 High level soil and waste samples are collected in glass containers (usually 125-mL clear glass) equipped with Teflon-lined caps. Soil samples may also be submitted as core samples contained in Encore samplers, metal or plastic "tubes", or in 40-mL VOA vials. The samples are iced at the time of collection and stored at 4C (less than 6C with no frozen samples). The holding time for soil and waste samples subjected to methanol extraction is14 days from date of collection; that is, the extraction and analysis must be completed within 14 days of collection.

5.4 TCLP leachate samples are collected with no headspace in Tedlar bags or syringes. The leachate samples are acidified at the time of collection (after the leaching procedure) with about 0.10mL of concentrated HCl per 40mL of sample and stored at 4C (less than 6C with no frozen samples) from the time leaching is completed until the analysis. The acidified leachate sample must be analyzed within 14 days of the leaching procedure. If the sample is not acidified, the leachate must be analyzed within 7 days of the leaching procedure.

NOTE: Samples that are suspected of having very high concentrations of VOC should be segregated from the "routine" samples and stored in a manner that will minimize sample and laboratory contamination. See STL-SL SOP AN70. If possible, keep the field QC in the same storage refrigerator as the samples.

6.0 APPARATUS AND MATERIALS

The apparatus and materials listed in this section may vary from lab to lab. The items listed are to give guidance and to provide a general overview of the equipment employed in this analysis.

6.1 Mass spectrometer: equipped with a capillary direct interface and a split/splitless injector or molecular jet separator

6.2 Gas chromatograph, compatible with the MS and purge and trap systems. If the GC is equipped with an injector that is operated in the split mode, the exit vent must have a carbon trap in-line to collect the volatile compounds that are vented during the transfer from the purge and trap device. The carbon traps should be changed a minimum of every three months.

6.3 Purge and trap device Tekmar 3000 Liquid Concentrator or equivalent

6.4 Supelco Vocarb 3000 trap or equivalent, Other traps may be used as long as the target compounds can be detected at the required quantitation limit.

6.5 Archon soil analyzer for low level soils, compatible with Tekmar purge and trap instruments. The instrument must be capable of automatically adding water and internal standard to the container while maintaining the septum seal, heating the sample to 40C, and spinning the stir bar to mix the sample during the purging step.

6.5 Data System compatible with the analytical system

6.6 Microsyringes: 10ul, 25ul, 50ul, 100ul, 250ul, 500ul, 2.5mL

6.7 Gastight syringe: 5mL, 25mL with luerlock tip

6.8 Volumetric flasks: 1.0mL, 10mL, 100mL

6.9 Recommended Columns

J&W DB-624: 60m x 0.32mm ID, 1.8um film
J&W DB-624: 20m x 0.18mm ID, 1.8um film

7.0 REAGENTS

Reagents must be tracked in accordance with STL-SL SOP AN44: *Reagent Traceability.*

7.1 Reagent water-free of volatile contaminants (obtained by purging with inert gas or carbon filtration)

7.2 Methanol-Burdich and Jackson, Purge and Trap grade

7.3 Sodium bisulfate-reagent grade. This salt is hydroscopic and should be stored in a dessicator.

7.4 Soil preservation solution- Slowly add, while stirring, 200g of sodium bisulfate to a 1.0-L volumetric containing about 700mL of reagent water. After the salt has dissolved, dilute to volume with reagent water, transfer to a storage container, and store the solution in an area free from VOC-especially water-soluble solvents such as acetone. The reagent should be tested prior to use by the analysis of a blank containing 5mL of the solution. The reagent is acceptable if it meets the same criteria as a method blank.

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions purchased from vendors or from stock standards prepared from neat materials. Certificates of analysis or purity must be received with all stock solutions or neat compounds. All preparation steps must be in accordance with STL-SL SOP AN41: *Standard Material Traceability.*

8.1 Preparation of **Stock Standards** from Neat Compounds

The lab should attempt to obtain a certified primary standard or secondary standard before preparing stock standards from neat materials. If primary stock standards must be prepared in-house, the target concentration range is from 2000ug/mL to 10000ug/mL. SL-SOP AN43: *Standard Preparation* gives the general instructions for the preparation of the stock solutions from neat materials.

8.2 Preparation of the **Working Standard** from Stock Standards

The working standard is prepared from the primary stock standards that are either prepared from neat compounds or purchased as certified solutions. The working standard contains one or more of the target compounds at a concentration suitable for preparing the calibration standards, generally 10-200ug/mL. A known volume of the working standard is then added to a known volume of reagent water to make the calibration standard.

The standards and standard concentrations listed in Table 1 are the suggested for routine use. If other "recipes" are used, the lab must document the standard preparation procedures in the standard traceability log.

8.3 Preparation of the **Calibration Standards** from the Working Standards

The calibration standards are the standards that are analyzed on the instrument. The calibration standard is made by adding a known volume of the working standard to a known volume of reagent water. The instrument must be calibrated using a minimum of five calibration standards. The lowest level standard must be at the reporting limit and the rest of the standards will define the working range of the analytical system.

8.3.1 Add 5.0mL of reagent water to a 5mL-glass syringe or 25ml of reagent water to a 25-ml glass syringe.

8.3.2 Add a known volume of the working standard to 5.0mL or 25ml of reagent water.

NOTE: The calibration standards for the low level soils are prepared using the same procedures as for the 5mL water purge except that the standards are purged at 40C. The lab has the option of using blank sand in the calibration standards.

The calibration standards listed in Table 1 are the suggested for routine use. If other "recipes" are used, the lab must document these standard preparation procedures in the standard traceablity log. A 5mL-purge volume may be used for low level (nominal RL of 1ug/L) if the instrument has sufficient sensitivity to detect the targets and the calibration criteria is met.

9.0 SAMPLE PREPARATION

Composite samples can be prepared using the guidance provided in STL-SL-SOP AN70.

9.1 **Aqueous samples** are analyzed directly by purge and trap/GC-MS. No sample preparation is necessary except to homogenize the sample prior to subsampling. The pH of liquid samples is checked and recorded prior to analysis to determine if the sample has been properly preserved.

9.2 Preparation of Soil Samples (5035)

9.2.1 Remove the Encore samples and the bulk sample from the storage area.

9.2.2 Test an aliquot of the bulk sample for the presence of carbonates.

- Transfer 5g of sample from the bulk sample to a 40mL vial..
- Add 5ml of the sodium bisulfate solution and shake the vial .
- If the sample exhibits effervescence, the Encore samples should be preserved as described above using 5mL of volatile-free water in place of the sodium bisulfate solution and placed in a freezer at –10C. The analytical hold time for frozen samples is 14 days from collection.
- If no effervescence is noted, the Encore samples may be preserved with 5mL soil preservation solution.

9.2.3 Add a stir bar to a vial and weigh the vial and record its tare weight(or tare the vial and stir bar weight by pressing the autotare button).

9.2.4 Transfer the sample from the Encore sampler to the tared vial and record the weight of the sample log.

If the sample effervesced during the carbonate test (9.2.2), add 5.0mL of reagent water and freeze at –10C. The hold time is 14 days from collection.

If not, add 5.0mL of the soil preservation solution, seal the vial, and store the sample at 4C until the time of analysis. The preserved sample must be analyzed within 14 days of collection.

NOTE: A preparation blank is prepared when Encore samples are transferred. The preparation blank contains the same reagents as the samples-either 5mL of reagent water or 5mL of soil preservation solution.

9.3 A methanol extraction is prepared when the concentration of the target compounds (by direct purge) exceeds the working range of the calibration curve. The bulk sample, collected in the 125-mL sample container, can be used to prepare the methanol extraction. Carry out the preparation quickly to minimize the loss of volatiles.

-Mix the sample with a stainless steel spatula and transfer 10g (+/- 0.5g) to a glass vial.

-Add 8uL of the surrogate spiking solution (2500ug/mL) to the sample and quickly add 10mL of purge and trap grade methanol. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 10g and 100% percent solids, is calculated:

$$Ct(ug/kg,dw) = \frac{0.008mL \otimes 2500ug/mL}{0.010g \otimes solids} = 2000ug/kg,dw$$

-Shake the sample for two minutes. Allow the solvent to separate from the solids portion of the sample and transfer a 1-2mL aliquot of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

-For each batch of twenty or fewer samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding 8uL of the surrogate spiking solution to 10mL of purge and trap grade methanol. Assume a sample weight of 10g. Analyze 125uL of the extract.

The lab control standard is prepared by adding 8uL of the surrogate spiking solution and 8uL of the matrix spiking solution to 10mL of purge and trap grade methanol. Assume a sample weight of 10g. Analyze 125uL of the extract.

The matrix spikes are prepared by adding 8uL of the surrogate spiking solution (2500ug/mL) and 8uL of the matrix spiking solution (2500ug/mL) to 10-g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for two minutes. Analyze 125uL of the extract or a smaller volume if the VOC concentration is high.

-Add 125uL of the extract (or a smaller volume if the VOC concentration exceeds the linear range of the system with 125uL) to 5.0mL of water (or to 25mL if the calibration is based on 25mL). Add the internal standard solution and analyze the sample using the ambient water calibration.

9.4 Methanol Extraction for Wastes

Carry out the preparation quickly to minimize the loss of volatiles.

9.4.1 Mix the sample with a stainless steel spatula and transfer 1g (+/- 0.2g) to a glass vial.

9.4.2 Add 10uL of the surrogate spiking solution (2500ug/mL) to the sample and quickly add 10mL of purge and trap grade methanol. If the sample is completely soluble in the methanol, dilute to a final volume of 10mL. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 1.0g , is calculated:

$$Ct(ug/kg) = \frac{0.010mL \otimes 2500ug/mL}{0.0010g \otimes solids} = 25000ug/kg$$

9.4.2 Shake the sample for one minute. Allow the solvent to separate from the solids portion of the sample and transfer 1mL to 2mL of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

For each batch of twenty or fewer samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding 8uL of the surrogate spiking solution (2500ug/mL) to 10mL of purge and trap grade methanol. Assume a sample weight of 1.0g. Analyze 100uL of the extract.

The lab control standard is prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) and 10uL of the matrix spiking solution (2500ug/mL) to 5.0mL of purge and trap grade methanol. Assume a sample weight of 5.0g. Analyze 100uL of the extract.

The matrix spikes are prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) and 10uL of the matrix spiking solution (2500ug/mL) to 1g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for one minute.

Add 100uL of the extract (or a smaller volume) to 5.0mL of water (or to 25mL if the calibration is based on 25mL). Add the internal standard solution and analyze the sample using the ambient water calibration.

NOTE: Waste samples may require significant dilution prior to analysis.

10.0 PROCEDURE

The following instrument conditions are recommended. The actual conditions may vary due to differences in instrumentation. The lab must document the instrument conditions in the maintenance log, the data system, or on the analysis log.

10.1 Instrument Conditions

10.1.1 GC Conditions
GC conditions may vary according to the environment and condition of each instrument. The lab must document the instrument conditions to assure consistent results and to aid in trouble-shooting the analytical system. Each lab is responsible for assuring that the conditions necessary to achieve adequate separation and sensitivity of the target analytes are maintained.

10.1.1.1 Example GC temperature program

Initial column temperature: 35 C for 3 minutes
Column temperature program 1: 20C per minute
Intermediate column temperature: 70C for 4 minutes
Column temperature program 2: 10C per minute
Final column temperature: 200C for 5.25 minutes

10.1.1.2 Column flow: Approximately 5-10mL/minute helium with a make-up of 20-25mL/minute helium. Total flow into the jet separator should be about 30mL/minute. The vacuum gauge on the jet separator will read about 0.5Torr.

If no jet separator is used and the column is plumbed directly into the source, the column flow should be adjusted to 0.5-1.0ml/min and a split ratio (desorb to column flow) of about 40:1 established. Smaller bore capillary columns (0.18 to 0.32mm) are required if the column is plumbed directly into the source

10.1.1.3 Mass Spectrometer and interface parameters

Jet separator temperature: 240C
Mass spectrometer interface: 240C
Mass spectrometer source temperature: factory set at 300C
range: 35-300amu, with a minimum scan cycle of 1 scan per second

10.1.2 Purge and Trap Conditions
The purge and trap conditions listed in this section are for guidance. The lab must document the actual conditions used. The purge time must be 11 minutes. Other parameters may be varied to optimize the detection of the target compounds.

10.1.2.1 "Three ring trap"-charcoal, Tenax, silica gel
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 180C
Bake time: 8 minutes at 225C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

10.1.2.1 VOCARB 3000 trap
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 225C
Bake time: 8 minutes at 250C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

The purge flow must be balanced for adequate sensitivity of the target compounds. If the purge flow is too high, the response of the gases will be low and not reproducible. The SPCC criteria for chloromethane may not be achieved if the purge flow is too high. If the purge flow is too low, the response of the more water-soluble targets-ketones, ethers, bromoform-may be low and the reporting limit may not be achieved on a routine basis.

10.2 BFB Tune Check

10.2.1 Fifty nanograms of 4-BFB must be analyzed at the beginning of each 12-hour clock as a check on the **"tune"** of the mass spectrometer. Meeting the tuning criteria ensures that the instrument is measuring the proper masses in the proper ratios. The 4-BFB analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of 4-BFB. All other instrument conditions must be identical-the mass range, scan rate, and multiplier voltage. If the instrument is configured for direct injection, 50ng of 4-BFB may be injected directly on to the column. If the purge and trap is used to analyze the 4-BFB, the purge and trap conditions must be the same as for the calibration standards and samples.

10.2.2 Evaluation of the 4-BFB peak.

10.2.2.1 The chromatogram should exhibit acceptable baseline behavior and the 4-BFB peak should be symmetrical. A spectrum of the baseline that shows high abundances of mass 40 (Argon) and mass 44 (carbon dioxide) may indicate a leak or contaminated carrier gas.

10.2.2.2 The spectrum of the 4-BFB must meet the criteria listed in the attached SOP Summary. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans +/- 5 scans from the apex can be evaluated for the 4-BFB criteria. Consecutive scans within this range can be averaged to meet the criteria.

10.2.2.3 The following records must be kept for each 4-BFB analysis that meets the criteria:
- the date, time, and data file of the analysis
- a spectrum of the scan or averaged scans
- a tabulation of the ion abundances of the scan

10.2.2.4 The 4-BFB analysis should be evaluated as to the relative size of the 4-BFB peak under the m/z 95 profile. A benchmark area window should be established for each instrument. Response outside of this window suggests instrumental problems such as a poor purge, clogged jet separator, leak in the Tekmar purging device, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open , or other anomalies.

10.2.2.5 If the 4-BFB fails to meet the acceptance criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the 4-BFB analysis, other corrective measures may include remaking the 4-BFB standard and/or cleaning the mass spectrometer source.

10.3 Initial Calibration

After the 4-BFB criteria has been met, the initial calibration standards are analyzed. Prepare the initial calibration standards according to the example recipes in the SOP appendices or lab-specific recipe. The lab must document the "recipe" used to prepare the calibration standards. The lowest level calibration standard must be at or below the routine RL and the other calibration standards will define the working range of the system.

10.3.1 Remove the plunger from the syringe and fill the barrel to overflowing with reagent water (syringe valve in the "red" position).

10.3.2 Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume(5mL or 25mL)

10.3.3 Briefly remove the syringe valve and inject the standards and internal standards into the syringe.

NOTE: Use the internal standard (IST) mix when preparing the calibration standards for analysis. The surrogates are already included in the standard mixes.

10.3.4 Load the standard(s) onto the purge and trap device and begin the analysis. All pertinent information concerning the standards must be recorded on the analysis log. The standards must be clearly identified and traceable to the preparation steps.

NOTE: The standards for low-level soil samples are prepared in the same manner as the 5mL standards. The standards for the low-level soils are purged at 40C. The lab has the option of using blank sand or soil in the calibration standards and the blank in the low level soil analysis.

10.3.5 After the acquisition has taken place, evaluate the calibration standards to ensure that each target compound, surrogate, and internal standard has been correctly identified. The analyst must be careful to complete this step before proceeding.

10.3.6 After each target compound, surrogate, and internal standard has been correctly identified, the relative response factor for each target compound and surrogate is calculated using the data system or using a PC spreadsheet as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where
Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (see the attached quantitation report for a list of the compounds that are associated with the various internal standards)
Cx = concentration or mass on-column of the target compound being measured (ug/L or ug/kg OR ng or ug on-column)
Cis = concentration or mass on-column of the internal standard (ug/L or ug/kg OR ng or ug on-column)

The average relative response factor (RRFavg) is calculated for each target compound and each surrogate compound:

$$RRFavg = \frac{RRF1 + RRF2 + ... + RRFn}{n}$$

where n = number of calibration levels

Calculate the standard deviation (SD) for the target compounds and surrogates at all calibration levels:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RFi - RFavg)^2}{n-1}}$$

where
Rfi = response factor of a target compound in the individual calibration level
Rfavg = average response factor
n= number of calibration levels

10.3.7 Calculate the relative standard deviation (% RSD) of the calibration levels for each target:

$$\%\,RSD = \frac{standard\ deviation}{RRFavg} \otimes 100$$

10.3.8 The results of the initial calibration are evaluated against the Calibration Check Compound (CCC) criteria and the System Performance Check Compound (SPCC) criteria, which are listed below. The CCC and SPCC criteria must be met before samples can be analyzed.

Calibration Check Compounds – CCC Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	Continuing Calibration
<=30% RSD	<=20% difference from initial calibration

System Performance Check Compounds-SPCC

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30 (0.10 for 25mL purge volume)

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

10.3.9 After the initial calibration criteria (CCC and SPCC) have been met, each target is evaluated for linearity.

If the %RSD of the target compound is less than or equal to 15%, the average response factor can be used for quantitation of samples.

If the %RSD of the target compound is greater than 15%, a regression curve (linear, quadratic, etc) must be used for the quantitation of samples. A regression curve may also be used for the compounds that have %RSD less than 15%. The results can be used to plot a calibration curve of response ratios-Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration or mass on-column of the target compound being measured (ug/L or ug/kg OR ng or ug)
Cis = concentration of the internal standard (ug/L or ug/kg OR ng or ug)

If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples.. Regression curves may be forced through zero but it is recommended that the curve be evaluated without forcing through zero first and then with the curve forced through the origin. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range

When more calibration levels are analyzed than required, individual compounds may be eliminated from the lowest or highest calibration levels(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of the calibration curve be eliminated without eliminating the entire level.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

8000B exception: evaluation of the "grand mean": If the average %RSD of ALL (all targets including CCC and SPCC) compounds in the initial calibration is less than 15%, the average response factor can be used for quantitation of all target compounds. The recommended course is to use regression curves, as described above, to quantify targets where the %RSD criterion (<=15%) is exceeded.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

10.3.10 After the initial calibration criteria has been met, the method blank is analyzed. 5.0mL or 25mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in Table 5 of the STL-SL CQAP or other specified QAP.

If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compounds must meet the criteria.

If the concentration of any target compound is above the RL in Table 5 of the STL-SL CQAP, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of samples can take place.

If the method blank repeatedly fails to meet the criteria, contact the immediate supervisor to determine the cause of the problem and to determine a course of action. This action may include re-cleaning the sparging tubes (with soap, hot water, and methanol), purging the effected autosampler ports with heated methanol, flushing the purge and trap ALS concentrator with methanol, replacing the trap, changing the transfer line, and changing the column. A method blank is then analyzed after taking the corrective action to demonstrate that the contamination has been eliminated. Once the system is determined to be free from contamination, sample analysis may begin. Method blanks may be required after the analysis of samples that contain very high levels of VOC.

10.4 Continuing Calibration Verification

At the beginning of each 12-hour clock, the tune of the instrument must be checked by the analysis of 50ng of 4-BFB. This criteria must be met before the analysis of the calibration check standards can take place.

10.4.1 After the tune criteria has been met, a continuing calibration check standard(s) is analyzed. The continuing calibration standard should be at a nominal concentration of 50ug/L-kg for 5ml/5g samples and 10ug/L for 25mL with ketones and poor purgeables at higher concentrations. The CCC and SPCC criteria (Section 10.3.8) must be met before the analysis of the method blank and samples can take place. The percent difference (%D) is calculated as follows:

$$\%D = \frac{RRFavg - RRFccv}{RRFavg} \otimes 100$$

where
RRFavg = average response factor from initial calibration
RRFccv = response factor from the check (12-hour) standard-calibration verification

The percent drift (%Drift) may also be used to evaluate the change/deviation of the curve:

$$\%Drift = \frac{Ci - Cccv}{Ci} \otimes 100$$

where
Ci = Calibration Check Compound standard concentration
Cccv = measured concentration using the selected quantitation method

NOTE: The SPCC criteria (10.3.8) must be met even if the regression curve option is used for quantitation. If this criteria is not met, corrective action must be taken. The corrective action may include reanalysis of the calibration check standard or preparation of a new secondary stock standard and reanalysis of the calibration check standard. If subsequent analysis of the standard is still out of criteria, a new initial calibration curve must be analyzed and evaluated.

10.4.2 The calibration standard (CCV) must also be evaluated for internal standard retention time and response.

If the retention time of any internal standard changes by more than 30 seconds from the retention times of the internal standards in the initial calibration, the analytical system must be inspected for problems and corrective action instituted.

If the extracted ion current profile (EICP) area for any of the internal standards changes by more than a factor of two (-50% to +100%) from the last calibration check standard, the analytical system must be inspected for problems and corrective action instituted. If the CCV is the first one after the initial calibration, compare the ISTD response to the corresponding level in the ICAL.

10.4.3 After the continuing calibration criteria has been met, the method blank is analyzed. 5.0mL or 25mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in Table 5 of the STL-SL CQAP.

If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compound must meet the criteria.

If the concentration of any target compound is above the RL in Table 5 of the STL-SL CQAP, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of client samples can take place.

10.5 Aqueous Sample Analysis-5.0mL to 25mL

The analyst must use the same volume as was used for the calibration standards-if a 5mL sample is used, it must be quanted off of the 5mL calibration curve; if a 25ml sample is used, it must be quanted off of the 25mL calibration curve. Samples are analyzed only after the tune criteria, the calibration (initial or continuing) criteria has been met, and the method blank criteria has been met. See the SOP Summary for the analytical sequence.

10.5.1 Remove the samples to be analyzed from the refrigerator and allow the samples to come to ambient temperature.

10.5.2 Put on a pair of gloves before transferring the sample from the vial to the syringe. The sample is most likely preserved with acid or may contain toxic or hazardous chemicals or biologically active components that may cause skin irritations. ***Gloves must be worn when handling samples.***

10.5.3 Mix the contents of the vial by inverting the vial several times. Check to see if there are air bubbles present in the sample. If air bubbles are present, use another vial if available. Make a note on the analysis log if the sample used contained bubbles and notify the supervisor and/or the project manager.

10.5.5 Remove the plunger from the glass syringe. Attach a syringe valve to the syringe Luer-tip to prevent sample from spilling out of the syringe when sample is added.

10.5.5 Open the vial of the well-mixed sample and gently pour the sample into the syringe barrel. The sample should fill the barrel of the syringe and overflow to allow trapped air bubbles to escape.

10.5.6 Replace the plunger into the syringe barrel. Try not to let air bubbles get into the barrel. If air bubbles are present, turn the syringe up, open the syringe valve , and expel the air while adjusting the volume to 5.0mL or 25mL. If no air bubbles were trapped, adjust the syringe to volume.

NOTE: For TCLP leachate samples, use 1.25mL of sample (1:4 dilution).

10.5.7 Open the syringe valve and inject the internal standard/surrogate (ISSU) mix into the sample.

10.5.8 Transfer the sample from the syringe to the purge and trap device. Record all of the sample identification information on the analysis log. Check the pH of the sample with pH paper and record the pH on the instrument log or other appropriate log.

10.5.9 Analyze the samples using the purge and trap and GC/MS conditions used for the initial and continuing calibration standards.

10.5.10 Determine the concentration of the samples and QC items. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

NOTE: Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client. For TCLP analyses, every reasonable effort should be made to achieve the regulatory level with out instrument overload.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with t the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower RLs.

A dilution is made when a volume of the sample is mixed with the reagent water to a final volume of 5.0mL or 25ml,depending on which curve is being used. The dilution factor is calculated by dividing the volume of sample into the volume used for the calibration curve.

$$DF = \frac{final\ volume\ of\ dilution(mL)}{volume\ of\ sample\ used(mL)}$$

For example, if 1.0mL of sample is diluted to final volume of 5.0mL, the dilution factor is 5. (5.0/1.0 = 5). If 1.0mL of sample is diluted to a final volume of 25mL, the dilution factor is 25 (25/1=25).

The following table gives some dilution factors:

Volume of Sample (mL)	Volume of Reagent Water (mL)	Final Volume (mL)	Dilution factor
5.0	0	5.0	1
2.5	2.5	5.0	2
1.0	4.0	5.0	5
0.5	4.5	5.0	10
0.10	4.9	5.0	50
25.0	0	25.0	1
5.0	20.0	25.0	5
2.5	22.5	25.0	10
1.0	24.0	25.0	25
0.50	24.5	25.0	50
0.10	24.9	25.0	250

NOTE: The same volume of internal standard/surrogate mix (ISSU) is added to the dilution as was added to the undiluted sample.

10.6 Low Level Soil Samples by Heated Purge and Trap (Method 5035)

The soil analytical system is calibrated using the same concentrations as the 5mL purge. The tune, initial and continuing calibration criteria, and the method blank criteria must be met before samples are analyzed. Standards and QC items must be analyzed under the same heated purge and trap conditions.

Remove the samples to be analyzed (Section 9.2) from the refrigerator or freezer and allow the sample to come to ambient temperature. Inspect the vial for cracks or obvious breaches in the septum. Load the samples on to the soil-purging unit and analyze according to the sequence described in Appendix B.

Liquid field QC for soils (trip blank, field blank, etc.) should be analyzed with the associated soil samples, using the same preparation and analytical procedures, including the heated purge. Report the results for liquid trip blanks as ug/L.

10.7 Analysis of Methanol Extracts of Soils and Wastes

The methanol extraction is used when the concentration of one or more target compounds exceeds the linear range of the low-level purge technique (>1000ug/kg). or if the concentration of VOC in the soil or waste samples is high. Samples are analyzed only after the 4-BFB criteria, the calibration criteria (initial and continuing), and the method blank criteria has been met. Medium level soil extracts are quanted using the ambient purge calibration curve. Sample preparation steps are included in Section 9.

10.7.1 Remove the plunger from the 5.0-mL syringe and fill the barrel to overflowing with reagent water(syringe valve in the "red" position). Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume(5mL)

10.7.2 Briefly remove the syringe valve and inject the sample extract and 5uL of the internal standard (IST) solution into the syringe. Use 125ul of the extract for soils and 100uL of the extract for wastes. Smaller aliquots are used if the concentration of target analytes exceed the working range of the system.

NOTE: Use the internal standard (IST) mix when preparing the medium level samples. Recall that the surrogates have already been added to the sample during the methanol extraction step (Section 9).

10.7.3 Load the sample on to the purge and trap device and begin the analysis. All pertinent information concerning the samples must be recorded on the analysis log. The samples must be clearly identified and traceable to the extraction log. These conditions must be the same as was used for the initial and continuing calibration standards-ambient purge for aqueous samples.

10.7.4 Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, a smaller aliquot of the methanol extract is reanalyzed to bring the highest target within the upper half of the calibration curve. Follow the guidelines in Section 10.4.10 for reporting dilutions.

NOTE: It is possible to dilute the surrogates in the sample extract below the linear range of the calibration curve. The minimum extract aliquot that can be used to provide a quantifiable result for the surrogates and matrix spikes is 0.0025mL (2.5uL).

SOIL: 10g to 10mL MeOH	WASTES: 1g to 10mL MeOH	Surrogates- Theoretical ng on-column
125uL(0.125mL)	100uL (0.100mL)	250
62.5uL(0.0625mL)	50uL(0.050mL)	125
25uL(0.025mL)	25uL(0.020mL)	50
12.5uL(0.0125mL)	10ul(0.010mL)	25
2.5uL(0.0025mL)	2.0uL(0.0020mL)	5.0-quantiation limit
<2.5uL(0.025mL)	<2.0uL(0.0020mL)	<5.0ng- below the quantitation limit-diluted out

NOTE: Some instrument quantitation limits may be higher than the limit listed in the table. The volume of extract should be adjusted accordingly.

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis of Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1 Two criteria must be met in order to identify a target compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{\textit{retention time of the target compound}}{\textit{retention time of the associated internal standard}}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. These ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.2 Tentatively Identified Compounds

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification. Tentative identifications of non-targets will be made only by analysts having completed the training specified in the training schedule.

11.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.2.2 The relative intensities of the major ions should agree within +/-30%.

11.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.

11.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

11.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria is not met exactly, the peak may be considered tentatively identified. The analyst should consult other analysts or the mass spectral interpretation specialist if there are any questions concerning an interpretation of spectra.

11.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculation is

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes DF$$

where
Cis = concentration of the internal standard, ug/L
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
DF = dilution factor

Soils by Heated P/T

$$TIC\,(ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{5.0g}{(W)(solids)}$$

where
Cis = concentration of the internal standard, ug/kg
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
W = weight of sample analyzed, g
solids = decimal equivalent of percent solids

Soils by Methanol Extraction

$$TIC\,(ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{Vcal}{(W)(solids)}$$

where
Cis = concentration of the internal standard, ug/kg
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = decimal equivalent of the percent solids(percent solids/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

where
Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)

11.3 Calculations for Samples-Internal Standard Technique

Aqueous Samples- relative response factor :

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes DF$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/L)
RRFavg = average response factor of the compound being measured
DF = dilution factor

Aqueous Samples: regression curve

$$concentration(ug/L) = concentration(curve) \otimes DF$$

where
DF = dilution factor

The reporting limit (RL) is calculated:

$$RL(ug/L) = RLqap \otimes DF$$

where
DF = dilution factor. The SL CQAP Table 5 RL(RLqap) assumes a DF of 1.

Soils by Heated P/T- relative response factor :

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{5.0g}{(W)(solids)}$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/kg)
RRFavg = average response factor of the compound being measured
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

Soils by Heated P/T: regression curve

$$conc(ug/kg,dw) = Ccurve(ug/kg) \otimes \frac{5.0g}{(W)(solids)}$$

where
Ccurve = concentration from curve(ug/kg)
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The reporting limit (RL) is calculated:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The STL-SL CQAP assumes W= 5.0g and solids = 1.

Methanol Extraction Soils and Wastes- relative response factor

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{Vcal}{(W)(solids)}$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/L)
RRFavg = average response factor of the compound being measured
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = (percent solids)/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)

Methanol Extraction of Soils and Solids- regression curve:

$$conc(ug,kg,dw) = Ccurve(ug/L) \otimes \frac{Vcal}{(W)(solids)}$$

where
Vcal = volume that calibration curve is based on (0.005L or 0.025L)
W = weight of sample added to the reagent water (g)-defined above

The reporting limit (RL)is calculated:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the reagent water (g)
solids = (percent solids)/100)

The STL-SL CQAP assumes W= 5.0g and solids = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in Section 3.1.3 of STL-SL SOP AN02: *Analytical Batching*. Note that the method blank for liquid samples and low-level soils is clock-specific and that the method blank for medium level soil samples is extraction batch-specific.

STL-SLSOP AN02: *Analytical Batching* describes the procedure for evaluating batch-specific QC. This criteria is summarized in the attached 8260 SOP Summary.

STL-SL SOP AN02 also contains the calculations for accuracy and precision and the calculations for the theoretical concentrations of surrogates, lab spikes, and matrix spikes.

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must demonstrate competence in the analysis of samples by this procedure. The minimum criteria for this demonstration is the preparation and analysis of spiked reagent water. Section 8.3 of EPA Method 8260A gives the general procedure for the performance of the IDOC and Table 6 of EPA Method 8260A gives the acceptance criteria for the accuracy and precision.

12.3 Method Detection Limit

The method detection limit is determined in accordance with STL-SL SOP CA90.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date. Section 10 of the STL-SL QAPs provide guidance on preventive maintenance.

14.0 TROUBLE-SHOOTING

Trouble-shooting items will be added at a later time. See instrument manufacturers' manuals for guidance on locating and repairing instrument problems.

15.0 REFERENCES

1. *Savannah Laboratories' Comprehensive Quality Assurance Plan* and *Savannah Laboratories' Corporate Quality Assurance Plan*, current revisions.
2. Method s 5035, 8000B, and 8260B. *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846.including Update III* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

Appendix A

VOLATILES BY GC/MS WORKING STANDARDS -EXAMPLE

These standards can be used to prepare the working standards for EPA Method 8260 to report the TCL (target compound list) compounds and the extended list of target compounds generally associated with EPA 8260. The standards are prepared in purge and trap grade methanol and are stored at 4C with minimum headspace.

Working Standard 1 (TCL WS-1)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA Cal #2	2000	12.5	25
VOA Cal #3	2000	12.5	25
VOA Cal #4	2000	12.5	25
1,2,-DCB	5000	5.0	25
1,3-DCB	5000	5.0	25
1,4-DCB	5000	5.0	25
2-CEVE	1000	125	125

Working Standard 2 (TCL WS-2)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA Cal #1	5000	25	125
8260 Surrogates	2500	10	25

Working Standard for GASES (TCL GASES)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
502.2 Cal 1	2000	12.5	25

Appendix A

Working Standard 3 (8260 WS-3)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
8260 Custom Mix #1	200	125	25
8260 Custom Mix #2	200	125	25
1,1,2,2-Tetrachloroethane	2000	12.5	25

Appendix A

Internal Standard (8260 ISTD)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
VOA ISTD	2500	20	50
1,2-DCE-d4	2000	25	50

Internal Standard/Surrogate (8260 ISSU)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA ISTD	2500	20	50
1,2-DCE-d4	2000	25	50
8260 Surrogate	2500	20	50

Tune Evaluation Standard (4-BFB)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
4-BFB	5000	10	50

Matrix Spike Standard (5-component subset)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
Matrix Spiking Solution	2500	20	50

TCLP matrix Spike Standard (5-component subset)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
TCLP Spiking Solution	2000	16	125

Appendix A

VOLATILES BY GC/MS CALIBRATION STANDARDS - EXAMPLES

The following calibration standards are prepared to define the working range of the EPA 8260 analysis for the target compound list (TCL) and the extended list of compounds generally associated with EPA 8260. The lowest level standard is at the reporting limit and the other standards define the working range. Samples with target analytes above the concentration of the highest calibration standard must be diluted and reanalyzed.

TARGET COMPOUND LIST

Working Level standards	Conc (ug/mL)	TCL-1 *	TCL-2 *	TCL-3 *	TCL-4 *	TCL-5 *	TCL-6 *
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 5.0mL of reagent water or to 5.0g of blank sand.

8260 EXTENDED LIST (TCL+ADDITIONAL COMPOUNDS)

Working Level standards	Conc (ug/mL)	8260-1 *	8260-2 *	8260-3 *	8260-4 *	8260-5 *	8260-6 *
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
8260 WS-3	25	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 5.0mL of reagent water or to 5.0g of blank sand.

CONCENTRATIONS OF THE CALIBRATION STANDARDS-5.0mL OR 5.0g

Cal Std	all targets except ketones, 2-CEVE	ketones, 2-CEVE
TCL-1,8260-1	5ug/l-kg	25ug/l-kg
TCL-2,8260-2	10ug/l-kg	50ug/l-kg
TCL-3,8260-3	25ug/l-kg	125ug/l-kg
TCL-4,8260-4	50ug/l-kg	250ug/l-kg
TCL-5,8260-5	100ug/l-kg	500ug/l-kg
TCL-6,8260-6	200ug/l-kg	1000ug/l-kg

Appendix A

VOLATILES BY GC/MS CALIBRATION STANDARDS-25mL Purge Volume-EXAMPLES

These calibration standards are prepared to define the working range of the EPA 8260 analysis for the target compound list (TCL) and the extended list of compounds generally associated with EPA 8260. The standards are based on a volume of 25mL to achieve lower quantitation limits for the target compounds. The lowest level standard is at the reporting limit and the other standards define the working range. Samples with target analytes above the concentration of the highest calibration standard must be diluted and reanalyzed.

TARGET COMPOUND LIST

Working Level standards	Conc (ug/mL)	25TCL -1*	25TCL-2*	25TCL-3*	25TCL-4*	25TCL-5*	25TCL-6*
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 25mL of reagent water.

8260 EXTENDED LIST (TCL+ADDITIONAL COMPOUNDS)

Working Level standards	Conc (ug/mL)	258260-1*	258260 -2*	258260-3*	258260-4*	258260-5*	258260-6*
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
8260 WS-3	25	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 25mL of reagent water.

CONCENTRATIONS OF THE CALIBRATION STANDARDS

Cal Std	all targets except ketones, 2-CEVE	ketones, 2-CEVE
25TCL-1,25-8260-1	1.0ug/l	5.0ug/l
25TCL-2,25-8260-2	2.0ug/l	10ug/l
25TCL-3,25-8260-3	5.0ug/l	25ug/l
25TCL-4,25-8260-4	10ug/l	50ug/l
25TCL-5,25-8260-5	20ug/l	100ug/l
25TCL-6,25-8260-6	40ug/l	200ug/l

Appendix B
8260 SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage*	Container	Hold Time
Aqueous	None; 4C	40mL no headspace	7 days
	HCl pH<2; 4C	40mL-no headspace	14 days
Soil/solid(low level)	Iced at collection; 5mL sodium bisulfate added upon arrival in lab; store at 4C	5-g Encore Sampler	14 days
Soil/solid(low level) -high carbonates	Iced at collection; 5mL water added upon arrival in lab; store at –10C	5-g Encore Sampler	14 days
Soil/solid(high level)	None; 4C	Glass 125mL	14 days
TCLP	HCl pH<2; 4C	Tedlar bag or syringe	14 days

*storage temperature is 4C with a control criteria of less than 6C with no frozen samples

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
4-BFB 50ng on column Clock starts at injection	4-BFB 50ng on column Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification (50ug/L or 50ug/kg)) RL Standard-low point on cal curve (if necessary)
Method blank	Method blank
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

See SL SOP AN02, Section 3.1.3, for the batch/clock options for LCS and MS/MSD.

Recommended Internal Standards:
1,2-dichloroethane-d4; 1,4-difluorobenzene; chlorobenzene-d5; 1,4-dichlorobenzene-d4

Surrogates/System Monitoring Compounds:
dibromofluoromethane; toluene-d8; 4-bromofluorobenzene

LCS/MS: CQAP Subset:
1,1-dichloroethene; benzene; trichloroethene; toluene; chlorobenzene

Appendix B
8260 SOP SUMMARY

VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION BROMOFLUOROBENZENE (BFB)	
m/e	**Abundance Criteria**
50	8.0-40.0% of mass 95
75	30.0-66.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	50-120%% of mass 95
175	4.0-9.0% of mass 174
176	93.0-101.0% of mass 174
177	5.0-9.0% of mass 176

(1) *8260 criteria taken from CLP OLMO4.0 (January 1998)

CALIBRATION ACCEPTANCE CRITERIA

Calibration Check Compounds - CCC
Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	Continuing Calibration
Less than or equal to 30% RSD	Less than or equal to 20% difference or drift from initial calibration

System Performance Check Compounds-SPCC

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30 (0.10 for 25mL purge volume)

See Sections 10.3 and 10.4 for ICAL and CCV linearity checks and criteria.

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
MS Tune Check - 50ng 4-BFB	Before initial and continuing calibration standards - every 12 hours	Mass abundances within method acceptance criteria	-Evaluate chromatogram and spectrum - Reanalyze - Retune MS and reanalyze - Remake standard and reanalyze - Perform instrument maintenance and reanalyze
Initial Calibration – minimum five point curve with lowest point at or below the Reporting Limit (RL)	Initially; after major instrument maintenance; whenever continuing calibration check fails. Prior to analysis of method blank and samples	Method criteria for CCC/SPCC (see -Calibration Acceptance Criteria – Table presented earlier in this document)	- Evaluate chromatograms, spectra, and integrations - Reanalyze standard(s) - Remake and reanalyze standard(s) - Perform instrument maintenance and recalibrate
Continuing Calibration check - midpoint standard	Every 12 hours before analysis of method blank and samples	Method criteria for CCC/SPCC (see Calibration Acceptance Criteria - Table presented earlier in this document)	- Evaluate chromatogram, spectra, integrations - Reanalyze standard - Remake and reanalyze standard - Recalibrate - Perform instrument maintenance and recalibrate
Method Blank	Every 12 hours (per clock) before sample analyses	All reported targets <RL	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
Lab Control Sample (LCS) -subset of target compounds unless full target spike specified by client	Each batch	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Matrix Spike/Matrix Spike Duplicate (MS/MSD) -subset of target compounds unless full target spike specified by client	Each batch	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Surrogates	All samples, blanks, LCS, MS	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Internal Standard Area	Evaluate all standards and samples	-Areas in continuing calibration verification must be 50% to +200% of previous initial calibration sequence -Retention time of internal standard must be +/-30 seconds from internal standard in initial calibration -Areas in samples should be evaluated for gross error . Consult supervisor.	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
Reporting Limit Standard -1x to 2x the RL	(Optional) Daily. Required for Florida DEP	Detected with reasonable response	-Evaluate chromatogram, spectra, and integrations -Reanalyze -Remake standard and reanalyze -Retune and recalibrate -Perform instrument maintenance and recalibrate
Initial Demonstration of Capability	Per analyst	Method criteria	-Reanalyze targets that do not meet criteria
Method Detection Limit (MDL)	See STL-SL SOP CA90	See STL-SL SOP CA90	-Reanalyze and re-evaluate

SEMI-VOLATILE COMPOUNDS BY GC/MS
Method: 8270C

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Approved by:

R. Wayne Robbins — 30 Aug 2002 (Date)

Title: Technical Manager

STL X Savannah __ Tallahassee __ Mobile __ Tampa West

1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine the concentration of various semi-volatile organic compounds (SVOC) in groundwater, TCLP and SPLP leachates, soils, sediments, wastes, and solid sample extracts. The attached quantitation report (Appendix B) lists the routine target compounds, the retention times of the target compounds, the characteristic ions of the target compounds, and the internal standard associated with each target compound.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits for the target compounds are given in Section 5 of the current revision of the Laboratory Quality Manual (LQM).

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using an appropriate extraction procedure. The extract is dried, concentrated to a volume of 1.0mL, and analyzed by GC/MS. Qualitative identification of the target compounds in the extract is based on the retention time and the mass spectra determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.2 This procedure is based on the guidance provided in SW-846 Method 8270C.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The toxicity or carcinogenicity of each chemical used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and lab glasses or face shield should be worn while handling extracts and standards. Standard preparation, addition of the internal standard solution, and sample extract dilution should be performed in a hood or well ventilated area.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst. These sheets specify the type of hazard that each chemical poses and the procedures that are used to handle these materials safely.

3.4 The exit vent of the splitless injector must have a carbon trap in-line to collect the semivolatile compounds that are vented during the injection of the extract. The traps should be changed every six months and disposed of in accordance with SOP CA70: *Waste Management*.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult. Elevated baselines may be due to oils, greases, or other hydrocarbons that may be extracted from improperly cleaned glassware or extraction vessels.

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. Sample extracts that contain high concentrations of non-volatile material such as lipids and high molecular weight resins and polymers may require the optional GPC cleanup prior to analysis. The GPC cleanup is generally not effective in removing non-target material that is associated with common petroleum products like diesel.

4.3 Secondary ions may be used for quantification if there is interference with the primary quantitation ion. If a secondary ion is used for quantification, the concentration/response relationship of the secondary ion must be established. The secondary ion must meet the same calibration criteria as the primary ion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days from TCLP leaching procedure	40 days

Refrigerator temperature acceptance criterion is less than 6C with no frozen samples.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph- Hewlett-Packard (HP) 5890 or equivalent with compatible autosampler, splitless injector, and direct capillary interface. The exit vent of the splitless injector must have a carbon trap in-line to collect the semivolatile compounds that are vented during the injection of extracts. The carbon traps should be changed every six months.

6.2 Mass spectrometer- HP5971, HP5972, HP5973 or equivalent

6.3 Recommended Capillary column-HP-5MS, 30m x 0.25mm ID x 0.25um film thickness or equivalent column

6.4 Data system- compatible with GC/MS system

6.5 Microsyringes- appropriate volumes

6.6 Volumetric flasks- Class A, appropriate volumes

6.7 Autosampler vials and crimper- compatible with autosampler

7.0 REAGENTS

Reagents must be tracked in accordance with SOP AN44: *Reagent Traceability.*

7.1 Methylene chloride- pesticide residue grade, for preparation of standards

7.2 Acetone- pesticide residue grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SOP AN41: *Standard Material Traceability.* General guidance on the preparation of standards is given in SOP AN43: *Standard Preparation.*

The lab should purchase certified solutions from STL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SOP AN43 for guidance for standard preparation from neat materials.

8.1 Preparation of the Stocks from Neat Standards

The steps for the preparation of primary stock standards from neat materials are given in SOP *AN43: Standard Preparation.* The standards should be prepared in methylene chloride but may require other solvents to dissolve the material.

8.2 Preparation the calibration standards from the stock standards

A minimum of five calibration standards are prepared. The concentrations of the stock standards are in the 1000-10000ug/mL range. The recommended standards are listed in Section 10.2. The lowest level standard should be at the equivalent of the reporting limit and the rest of the standards should define the working range of the detector. Note that six calibration levels are required for a second order regression curve. Internal standards should be added to each standard to give a final concentration of 40ug/mL.

Each lab should develop controlled recipes that can be posted or maintained in appropriate logbooks.

9.0 SAMPLE PREPARATION

9.1 The sample extraction procedures are given in the following SOPs:

Matrix	SOP	Extraction Technique
Aqueous, TCLP leachates	EX30	Continuous Liquid-liquid Extraction
Aqueous, TCLP leachates	EX35	Separatory Funnel
Soils/Sediments	EX40	Sonication
Wastes	EX42	Waste dilution

9.2 The sample concentration procedures are given in SOP EX 50: Zymark Nitrogen Concentration.

9.3 Gel permeation chromatography (SOP EX61) may help to eliminate or minimize matrix interferences in a limited number of samples. The GPC cleanup is generally not effective on samples containing petroleum products.

10.0 PROCEDURE

10.1 Instrument Conditions

Instrument conditions may vary according to the sensitivity of each instrument. The following conditions are provided for guidance. The lab must optimize and document the conditions used for the analysis of SVOC by GC/MS.

Recommended Column:

HP-5MS 30m x 0.25mm ID x 0.25um film thickness or equivalent

Column flow: Approximately 1mL/min helium

GC Oven temperatures:

Initial column temperature: 45 C for 3 minutes
Column temperature program: 10C per minute
Final column temperature: 300C (until at least one minute past the elution time of Benzo (g,h,i) perylene).

GC injector parameters

Injector temperature: 250-270EC
Injection type: split, approximately 1:10 or splitless injection
Injector liner: 4mm ID quartz or 4mm glass, deactivated (single "Gooseneck")
Sample injection volume: 1-2uL

Mass Spectrometer and interface parameters

Mass spectrometer interface: 300C
Mass spectrometer source temperature: Factory Set
Mass range: 35-500amu, with a scan time of 1.0 scans per second or greater

10.2 Calibration

A minimum of five calibration standards are prepared and analyzed. The recommended standards are 10, 20, 50, 80, 100, 200ug/mL. The lowest level standard should be at or below the equivalent of the reporting limit and the rest of the standards should define the working range of the detector. Note that six calibration levels are required for a second order regression curve.

10.2.1 Fifty nanograms of DFTPP must be analyzed at the beginning of each 12-hour clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria demonstrates that the instrument is measuring the proper masses in the proper ratios. The DFTPP analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of DFTPP. All other instrument conditions must be identical-the mass range, scan rate, and multiplier voltage.

10.2.1.1 Prepare a 50ng/uL solution of tune/column evaluation standard containing each of the following compounds at 50ug/mL in methylene chloride: DFTPP, pentachlorophenol, p,p'-DDT, and benzidine.

10.2.1.2 Analyze a 1uL aliquot of the tune/column evaluation solution.

10.2.1.3Evaluate the DFTPP peak.

-The chromatogram should exhibit acceptable baseline behavior and the DFTPP peak should be symmetrical.

-The spectrum of the DFTPP must meet the criteria listed in the SOP Summary (Appendix A). Background subtraction must be straightforward, that is, no scan within the elution window of DFTPP may be subtracted from another scan within the elution window, and designed only to eliminate column bleed or instrumental background. Scans +/- 2 scans from the apex can be evaluated for the DFTPP criteria. Consecutive scans within this range may be averaged to meet the criteria.

NOTE: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

10.2.1.4Benzidine and pentachlorophenol should be present at their normal responses with minimal peak tailing visible. Peak tailing guidance is taken from EPA Method 625 which allows pentachlorophenol to be less than or equal to five and benzidine less than or equal to three. Refer to Figure 1for an example of [eak tailing factor calculation.

This is a good check on the system: if pentachlorophenol (a CCC) does not respond well, the calibration standard should not be analyzed. Injector port and column maintenance should be performed and the tune/column evaluation standard reanalyzed.

The percent breakdown of p,p'- DDT is calculated using the following equation. The percent breakdown should not exceed 20%.

$$\%Breakdown = \frac{(areaDDE + areaDDD)}{(areaDDT + areaDDE + areaDDD)} x\ 100$$

Areas from the total ion chromatogram are used to calculate DDT breakdown.

10.2.2 After the DFTPP criteria and column evaluation criteria have been met, the initial calibration standards are analyzed.

10.2.2.1Prepare the initial calibration standards. The lowest calibration standard should be at the RL and the rest of the standards will define the working range. See section 10.2 for guidance regarding calibration levels.

10.2.2.2Set up a sequence and analyze the calibration standards. The injection volume must be the same for the calibration standards and all sample extracts.

10.2.3 Identify the internal standards, surrogates, and the target compounds. The data system must be updated with the proper retention times and ion data.

10.2.4 Calculate the relative response factor for each compound as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured
(See the attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration of the compound being measured (ug/mL)
Cis = concentration of the internal standard (40ug/mL)

Secondary ions may be used for quantification if there is interference with the primary quantitation ion. If a secondary ion is used for quantification, the concentration/response relationship of the secondary ion must be established. The secondary ion must meet the same calibration criteria as the primary ion.

10.2.5 Calculate the average relative response factor (RRF_{avg}) for each target compound and each surrogate compound:

$$RRF_{avg} = \frac{RRF1 + RRF2 + RRF3.... + RRFn}{n}$$

RRF1 = relative response factor of the first standard
RRFn = relative response factor of the last standard
n = number of calibration standards

10.2.6 Calculate the standard deviation (SD) for the initial calibration standards:

$$SD = \sqrt{\frac{\sum_{i-1}^{n_l} (RRF_i - RRF_{avg})^2}{n-1}}$$

10.2.7 Calculate the relative standard deviation (%RSD) of the target compounds in the calibration standards.

$$\%RSD = \frac{SD}{RRF_{avg}} x\ 100$$

10.2.8 Evaluation of the Initial Calibration

The initial calibration is evaluated specifically for the calibration check compounds (CCC) and the system performance check compounds (SPCC). The CCC and SPCC criteria are given in the SOP Summary (Appendix A). The %RSD criteria for CCC and minimum RRF for SPCC must be met before the analysis of sample extracts can begin.

If the CCC and SPCC criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the initial calibration must be repeated. The analyst must be aware of the 12-hour clock for the DFTPP analysis. The DFTPP criteria must be met prior to the analysis of the calibration standards.

10.2.9 After the initial calibration criteria (CCC/SPCC) have been met, each target is evaluated for linearity. Refer to SOP AN67: *Evaluation of Calibration Curves* for guidance.

If the %RSD of the target compound is less than or equal to 15%, the average response factor can be used for quantitation of samples.

If the %RSD of the target compound is greater than 15%, a regression curve (linear, quadratic, etc) must be used for the quantitation of samples. A regression curve may also be used for the compounds that have %RSD less than 15%. The results can be used to plot a calibration curve of response ratios-Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where:

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See attached quantitation report for a list of the compounds and their associated internal standard)
Cx = concentration of the target compound being measured (ug/mL)
Cis = concentration of the internal standard (ug/mL)

A linear or quadratic curve may be used to define the concentration/response relationship. If r^2 is greater than 0.99, the curve can be used to quantify samples. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire concentration range.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

When more calibration levels are analyzed than required, individual compounds may be eliminated from the lowest or highest calibration levels(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of the calibration curve be eliminated without eliminating the entire level.

8000B exception: evaluation of the "grand mean": If the average %RSD of ALL (all targets including CCC and SPCC) compounds in the initial calibration is less than 15%, the average response factor can be used for quantitation of all target compounds. The recommended course is to use regression curves, as described above, to quantify targets where the %RSD criterion (<=15%) is exceeded.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

10.3 Continuing Calibration Verification

At the beginning of each 12-hour clock, the tune of the instrument must be checked by the analysis of the tune/column evaluation solution (10.2.1.1). The tune and column evaluation criteria (10.2.1.3 and 10.2.1.4) must be met before the analysis of the calibration check standards can take place.

10.3.1 After the tune and column evaluation criteria have been met, a continuing calibration check standard(s) is analyzed. The continuing calibration standard should be at a mid-level concentration. The CCC and SPCC criteria (SOP Summary, Appendix A) must be met before the analysis of samples can take place. The percent difference (%D) is calculated as follows:

$$\%D = \frac{RRFavg - RRFccv}{RRFavg} \otimes 100$$

where
RRFavg = average response factor from initial calibration
RRFccv = response factor from the check (12-hour) standard-calibration verification

The percent drift (%Drift) may also be used to evaluate the change/deviation of the curve:

$$\%Drift = \frac{Ci - Cccv}{Ci} \otimes 100$$

where
Ci = Calibration Check Compound standard concentration (ug/mL)
Cccv = measured concentration using the selected quantitation method (ug/mL)

NOTE: The SPCC criteria (10.3.8) must be met even if the regression curve option is used for quantitation. If these criteria are not met, corrective action must be taken. The corrective action may include reanalysis of the calibration check standard or preparation of a new secondary stock standard and reanalysis of the calibration check standard. If subsequent analysis of the standard is still out of criteria, a new initial calibration curve must be analyzed and evaluated.

10.3.2 The continuing calibration verification standard (CCV) must also be evaluated for internal standard response.

If the extracted ion current profile (EICP) area for any of the internal standards in the CCV changes by more than a factor of two (-50% to +100%) from the last initial calibration sequence, the analytical system must be inspected for problems and corrective action instituted.

10.4 Samples are analyzed only after the DFTPP criteria, column evaluation criteria and the calibration verification criteria have been met. The analytical system must be evaluated every 12 hours by the analysis and evaluation of the tune/column evaluation standard and a mid-level calibration standard.

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
Tune/Column Evaluation Standard Clock starts at injection	Tune/Column Evaluation Standard Clock starts at injection
Calibration standards- Minimum of five cal levels	Mid point calibration verification Optional RL: Standard-low point on cal curve
Samples analyzed until 12-hour clock expires	Samples analyzed until 12-hour clock expires

10.4.1 Remove the sample extracts to be analyzed from the refrigerator and allow the sample to come to ambient temperature.

10.4.2 Add 20-uL of the internal standard mix (2000 ug/mL) to each 1.0mL aliquot of the sample extract. The concentration of the internal standard in the extract is 40 g/mL.

10.4.3 Mix the contents of the autosampler vial by inverting several times.

10.4.4 Analyze the samples using the same analytical conditions used for the initial and continuing calibration standard. Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

NOTE: Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client. For TCLP analyses, every reasonable effort should be made to achieve the regulatory level with out instrument overload.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with t the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower RLs.

10.4.5 The dilution factor is calculated by dividing the volume of sample extract in microliters into 1000. For example, if 100uL of a sample extract are diluted to final volume of 1.0mL, the dilution factor is 10. (1000/100 = 10). The following table gives some dilution factors:

Dilution Preparation

uL extract-Vext	uL MeCl2	volume of dilution (Vdil-uL)	uL ISTD (2000ug/mL)-Vistd	DF
1000	0	1000	20	1
500	500	1000	10*	2
200	800	1000	16*	5
100	900	1000	18*	10
50	950	1000	19*	20
20	980	1000	20*	50

*assumes dilution of a 1mL extract or 1mL aliquot of an extract that has been spiked with the internal standard at 40ug/mL using 20ul of a 2000ug/mL internal standard solution

The concentration of internal standards must remain constant for all extracts and extract dilutions at 40ug/mL. The following equation can be used to determine the volume of the 2000ug/mL internal standard solution to add to an extract when a dilution is prepared from an extract that has already been spiked with the internal standard solution:

$$Vistd(uL) = 20uL - \left(\frac{Vext}{Vdil} \otimes 20ul\right)$$

Vistd = volume of 2000ug/mL internal standard to add to the diluted extract (uL)
Vext = volume of extract used to prepare the dilution (uL)
Vdil = final volume of the dilution (uL)-1000uL (1.0mL)

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis

11.1.1 Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from the daily calibration standard or a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1.1 Two criteria must be met in order to positively identify a compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{\textit{retention time of the target compound}}{\textit{retention time of the associated internal standard}}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. Ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.1.2 Tentatively Identified Compounds (TICs)

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification.

The default procedure is to evaluate up to 20 compounds of greatest apparent concentration that are not included as target compounds or routinely reported volatile compounds. The unknown compounds are tentatively identified using a forward search of the reference library.

If the library search produces a match at or above 85%, report that compound. If the library search produces more than one compound at or above 85%, report the first compound (the highest match quality). If the library search produces no matches at or above 85%, report the compound as unknown. If possible, provide a general classification of the unknown – for example, unknown aromatic, unknown hydrocarbon, etc.

TICs should be evaluated within the retention time range from the first eluting target or surrogate (whichever is first in the target list) to three minutes after the elution of the last target compound.

11.1.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.1.2.2 The relative intensities of the major ions should agree within +/-20%.

11.1.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.

11.1.2.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

11.1.2.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.1.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria is not met exactly, the peak may be considered tentatively identified. The analyst should consult senior analysts or the mass spectral interpretation specialist if there are any questions concerning an interpretation of spectra.

11.1.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculations assume that the same volume is injected for standards and samples.

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{F}{V} \otimes DF$$

where:

Cis =	concentration of the internal standard (ug/mL)
AREAis =	total ion peak area of the internal standard
AREAtic=	total ion peak area of the TIC
F =	final volume of extract (mL)
V =	volume of sample extract (L)
DF =	dilution factor

Soils

$$TIC\ (ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{F}{(W)(solids)} \otimes DF$$

where:

Cis =	concentration of the internal standard, ug/mL
AREAis =	total ion peak area of the internal standard
AREAtic=	total ion peak area of the TIC
F =	final volume of extract mL
W =	weight of sample analyzed (kg)
solids =	decimal equivalent of percent solids

11.2 Calculations for Samples-Internal Standard Technique
These calculations assume that the same volume is injected for standards and samples and that the standards and samples have the same concentration of internal standard.

11.2.1 Aqueous Samples

11.2.1.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{V} \otimes DF$$

where:

Ax =	area of the characteristic ion of the compound being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

11.2.1.2 If a regression curve is used, the concentration is given:

$$concentration(ug/L) = Ccurve \otimes \frac{F}{V} \otimes DF$$

where:

C_{curve} =	concentration from curve (ug/mL)
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

11.2.1.3 The reporting limit (RL) for each sample is given:

$$RL(ug/L) = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Vqap}{V} \otimes DF$$

where:

F = final volume of extract (mL)
Fqap = 1.0mL
Vqap = 1.0L
V = volume of sample extracted
DF = dilution factor. The LQM RL assumes a DF of 1.

NOTE: If V = 800mL to 1200mL, assume that Vqap/ V = 1 in the calculation of the reporting limit.

11.2.2 Soils

11.2.2.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ax =	area of the characteristic ion of the compound being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
W =	weight of sample extracted (kg)
solids =	(percent solids)/100
DF =	dilution factor

11.2.2.2 If the regression curve is used, the concentration is given:

$$conc(ug/kg,dw) = Ccurve \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ccurve =	concentration from curve(ug/mL)
W =	weight of sample extracted (kg)
F =	final volume of extract (mL)
solids =	(percent solids)/100)
DF =	dilution factor

11.2.2.3 The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Wqap}{(W)(solids)} \otimes DF$$

where

F = final volume of extract (mL)
W = weight of sample extracted (kg)
solids = (percent solids)/100

The LQM assumes Wqap = 30g, solids = 1, Fqap = 1.0mL, and DF = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in AN02: *Analytical Batching.* SOP AN02 also describes the procedure for evaluating batch-specific QC. The QA/QC criteria are summarized in the SOP Summary (Appendix A).

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must participate in the analysis of samples by this procedure in accordance with SOP CA92: *Evaluation of IDOCs.*

12.3 Method Detection Limit

The method detection limit is determined in accordance with SOP CA90: *Procedure for the Determination of the Method Detection Limit.*

13.0 PREVENTIVE MAINTENANCE & TROUBLESHOOTING

Refer to SOP AN53: *Preventive Maintenance Procedures for Laboratory Instruments* for guidance.

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Refer to SOP CA70: *Waste Management* for proper waste handling procedures.

15.0 REFERENCES

15.1 *STL Savannah Laboratory Quality Manual* current revision.

15.2 Method 8270C:*Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

APPENDIX A
8270C SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days	40 days

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
Tune/Column Evaluation Standard Clock starts at injection	Tune/Column Evaluation Standard Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification standard RL Standard (lowest point on calibration curve if required by client or state-specific QAP)
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION (DFTPP)	
m/e	**Ion Abundance Criteria (1)**
51	30-80% of mass 442
68	Less than 2.0% of mass 69
69	Present
70	Less than 2.0% of mass 69
127	25-75% of mass 198
197	Less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5.0-9.0% of mass 198
275	10-30% of mass 198
365	Greater than 0.75% of mass 198
441	Present but less than mass 443
442	40-110% of mass 198
443	15.0-24.0% of mass 442

(1) 8270 criteria taken from CLP OLMO4.0 (January 1998). The use of alternate criteria is expressly allowed in SW-846 Method 8270C.

APPENDIX A
8270C SOP SUMMARY

CALIBRATION ACCEPTANCE CRITERIA

Calibration Check Compounds - CCC
Phenol, 1,4-Dichlorobenzene, 2-Nitrophenol, 2,4-Dichlorophenol, Hexachlorobutadiene, 4-Chloro-3-methylphenol, 2,4,6-Trichlorophenol, Acenapthene, N-Nitrosodiphenylamine, Pentachlorophenol, Fluoranthene, Di-n-octylphthalate, Benzo(a) pyrene

System Performance Check Compounds-SPCC
N-Nitrosodi-n-propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol, 4-Nitrophenol

Initial Calibration	Continuing Calibration*
CCC: <= 30% RSD SPCC: RRFavg >= 0.050	CCC: <= 20% difference from initial calibration SPCC: RRF>= 0.050

*If CCC and/or SPCC do not meet the stated criteria, all targets that are reported must meet the CCC criteria.

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

The results for all target compounds are evaluated for linearity. If the %RSD is less than 15%, the calibration is assumed linear through the origin and the average response factor can be used for quantitation. If the average response factor for the target exceeds 15% (including any CCC), the analyst must use the calibration curve option.

NOTE: The lab has the option of using a regression curve for all analytes.

A linear, quadratic, or higher order regression fit may be used to define the concentration/response relationship. If r^2 is greater than 0.99, the curve can be used to quantify samples. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range. The minimum number of calibration standards required for a regression curve are given in the following table:

Type of curve	Minimum Number of Calibration Points
Linear (first order)	5
Quadratic (second order)	6

QC Item	Frequency	Acceptance Criteria	Corrective Action
Tune/Column Evaluation Standard DFTPP 50ng Pentachlorophenol - 50ng Benzidine – 50ng p,p'-DDT 50ng	Prior to analysis of calibration standards every 12 hours	DFTPP - within criteria	-Evaluate alternative scans -Reanalyze and evaluate -Retune and reanalyze -Clean source, retune, reanalyze
		Pentachlorophenol and benzidine - present at usual response with no peak tailing visible p,p'-DDT - %breakdown <20%	-Reanalyze -Perform injector port maintenance and reanalyze -Cut more than usual length of column and reanalyze -Replace column
Initial Calibration	After Tune Check and when calibration verification standard fails acceptance criteria. All initial calibration standards	CCC: %RSD < 30% SPCC: RRFavg > 0.050 Use regression curve for quantitation if %RSD for any target compound exceeds 15%	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Continuing Calibration Verification	After tune check; every 12 hours prior to analysis of samples	CCC: %Difference <= 20% Or %Drift <= 20% SPCC: RRF >= 0.050	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Internal Standard Areas	Evaluate all standards and samples	Areas in continuing calibration verification must be 50% to +200% of previous initial calibration sequence Areas in samples should be evaluated for gross error. Consult supervisor Retention time of internal standard must be +/-30 seconds from internal standard in previous CCV.	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available -Recalibrate

QC Item	Frequency	Acceptance Criteria	Corrective Action
Surrogate recovery	Evaluate for all samples and QC items if extract is not diluted OR If diluted, where >RL	Within LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Method Blank	Per batch	All targets < RL in LQM	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
Lab Control Standard (LCS) - QAP subset	Per batch See SOP AN02	Within LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
Matrix spike (MS) Matrix spike duplicate (MSD)	Per batch if sufficient sample volume/weight supplied See SOP AN02	Within LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
RL Standard (reporting limit)	Daily (optional)-lowest point on calibration curve if required by client or state-specific QAP	Detected at reasonable sensitivity	-Evaluate integrations and spectra; -Reanalyze -Prepare new standard and reanalyze
Initial Demonstration of Capability (IDOC)	Each work group	Accuracy and precision within method specified criteria	-Evaluate data -Reanalyze extracts if warranted -Re-extract and reanalyze for targets that fail criteria
Method Detection Limit (MDL)	Annually for each routine matrix See SOP CA90	Evaluate according to SOP CA90	Evaluate according to SOP CA90

APPENDIX B- TARGET COMPOUNDS

ROUTINE TARGET LIST

PARAMETER	RT	Quant Ion	Secondary Ions		ISTD
1,4-Dioxane	1.894	88	58	45	1
Pyridine	2.123	79	52	51	1
N-Nitrosodimethylamine	2.102	42	74		1
Aniline	3.812	93	66		1
Phenol	3.796	94	66	65	1
Bis(2-chloroethyl)ether	3.854	63	93	95	1
2-Chlorophenol	3.908	128	130	64	1
1,3-Dichlorobenzene	4.025	146	148	111	1
1,4-Dichlorobenzene	4.073	146	148	111	1
Benzyl Alcohol	4.202	108	79	77	1
1,2-Dichlorobenzene	4.239	146	148		1
2-Methylphenol	4.314	107	108	77	1
bis(2-Chloroisopropyl)ether	4.335	45	121		1
N-Nitroso-di-n-propylamine	4.469	70	42	101	1
3&4-Methylphenol	4.447	107	108		1
Hexachloroethane	4.522	117	201	199	1
Nitrobenzene	4.602	77	123	65	2
Isophorone	4.837	82	95	138	2
2-Nitrophenol	4.923	139	109	65	2
2,4-Dimethylphenol	4.965	107	122	121	2
Bis(2-chloroethoxy)methane	5.067	93	123	95	2
Benzoic acid	5.115	105	122		2
2,4-Dichlorophenol	5.169	162	164	98	2
1,2,4-Trichlorobenzene	5.259	180	182	145	2
Naphthalene	5.323	128	129		2
4-Chloroaniline	5.409	127	129	65	2
Hexachlorobutadiene	5.532	225	223	227	2
4-Chloro-3-methylphenol	5.991	107	144	142	2
2-Methylnaphthalene	6.135	142	141		2
1-Methylnaphthalene	6.269	142	141		2
Hexachlorocyclopentadiene	6.429	237	235	272	3
2,4,6-Trichlorophenol	6.541	196	198	200	3
2,4,5-Trichlorophenol	6.590	196	198	200	3
2-Chloronaphthalene	6.760	162	164	127	3
2-Nitroaniline	6.958	65	92	138	3
Dimethylphthalate	7.268	163	194	164	3
2,6-Dinitrotoluene	7.353	165	89	63	3
Acenaphthylene	7.337	152	151	153	3
3-Nitroaniline	7.540	138	108	92	3
Acenaphthene	7.599	154	153	152	3
2,4-Dinitrophenol	7.685	184	63	154	3
4-Nitrophenol	7.824	65	109	139	3
Dibenzofuran	7.829	168	139		3
2,4-Dinitrotoluene	7.914	165	89	63	3
2,3,4,5-Tetrachlorophenol	8.064	232	230	131	3
2,3,4,6-Tetrachlorophenol	8.091	232	230	131	3
Diethylphthalate	8.310	149	177	150	3

Fluorene	8.336	166	165	167	3
4-Chlorophenyl-phenylether	8.363	204	141	206	3
4-Nitroaniline	8.454	138	108	92	3
4,6-Dinitro-2-methylphenol	8.513	198	105	121	4
N-Nitrosodiphenylamine	8.555	169	168	167	4
1,2-Diphenylhydrazine	8.593	77	105	182	4
4-Bromophenyl-phenylether	9.090	248	250	141	4
Hexachlorobenzene	9.293	284	142	249	4
Pentachlorophenol	9.581	266	264	268	4
Phenanthrene	9.784	178	176	179	4
Anthracene	9.854	178	176	179	4
Carbazole	10.137	167			4
Di-n-Butylphthalate	10.847	149	150	104	4
Fluoranthene	11.659	202	203	101	4
Benzidine	11.926	184	92	185	5
Pyrene	12.006	202	200	203	5
Butylbenzylphthalate	13.214	149	91	206	5
3,3'-Dichlorobenzidine	13.892	252	254	126	5
Benzo(a)Anthracene	13.866	228	229	226	5
Bis(2-ethylhexyl)phthalate	14.111	149	167	279	5
Chrysene	13.924	228	226	229	5
Di-n-octylphthalate	14.971	149	43		5
Benzo(b)fluoranthene	15.367	252	253	125	6
Benzo(k)fluoranthene	15.399	252	253	125	6
Benzo(a)pyrene	15.783	252	125	253	6
Indeno(1,2,3-cd)pyrene	17.284	276	139		6
Dibenzo(a,h)anthracene	17.317	278	139	279	6
Benzo(g,h,i)perylene	17.674	276	277	138	6
SURROGATES					
2-Fluorophenol	3.032	112	64		1
Phenol-d5	3.785	99	71		1
Nitrobenzene-d5	4.586	82	128	54	2
2-Fluorobiphenyl	6.643	172	171		3
2,4,6-Tribromophenol	8.732	330	332	141	3
Terphenyl-d14	12.332	244	122	212	5
INTERNAL STANDARDS					
1,4-Dichlorobenzene-d4	4.057	152	150	115	1
Naphthalene-d8	5.302	136	68		2
Acenaphthene-d10	7.556	164	162	160	3
Phenanthrene-d10	9.747	188	94	80	4
Chrysene-d12	13.887	240	236	120	5
Perylene-d12	15.858	264	265	260	6

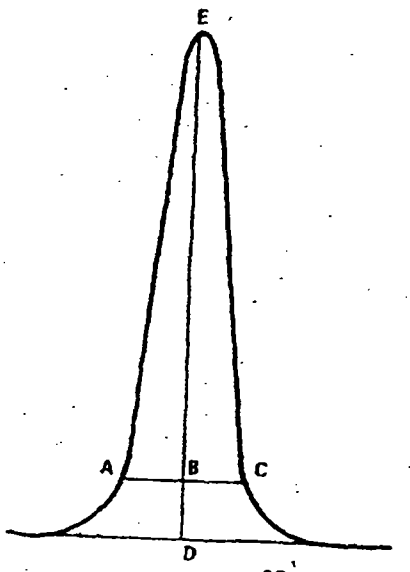
APPENDIX B- TARGET COMPOUNDS

APPENDIX IX TARGET LIST

PARAMETER	RT	Quant Ion	Secondary Ions			ISTD
1,4-Dioxane	1.933	88	58	45		1
Pyridine	2.178	79	52	51		1
2-Picoline	2.664	93	66			1
1,4-Benzoquinone	4.466	54	108	82		1
N-Nitrosomethylethylamine	2.739	88	42	43	56	1
Methyl methanesulfonate	2.964	80	79	65		1
N-Nitrosodiethylamine	3.268	102	42	44	57	1
Ethyl methanesulfonate	3.503	79	109	97	45	1
N-Nitrosopyrrolidine	4.481	100	41	42		2
Acetophenone	4.486	105	77	51		2
N-Nitrosomorpholine	4.497	56	86			1
O-Toluidine	4.529	106	107	79		2
Phorate	9.123	75	121			4
N-Nitrosopiperidine	4.796	114	42	55	56	2
O,O,O-Triethyphosphorothioate	5.122	65	97	93		2
2,6-Dichlorophenol	5.469	162	164	98		2
Hexachloropropene	5.507	213	211	215	117	2
1,2,4,5-Tetrachlorobenzene	6.447	216	214	179		3
a-Dimethylphenethylamine	5.619	58	91	42		2
-Nitroso-di-n-butylamine	5.875	84	57	41		2
1,4-Phenylenediamine	5.864	108	80	107		2
Safrole	6.094	162	104	135	103	2
Isosafrole	6.757	162	104	131		2
1,1-Biphenyl	6.805	154	76			3
1,4-Naphthoquinone	7.056	158	104	76		3
m-Dinitrobenzene	7.317	168	76	50		3
Pentachlorobenzene	7.916	250	248	252	215	3
1-Naphthylamine	8.001	143	115	116		3
2-Naphthylamine	8.113	143	115	116		3
2,3,4,6-Tetrachlorophenol	8.140	232	230			3
5-Nitro-o-toluidine	8.466	152	77	106		3
Thionazin	8.471	107	96	97		4
Sulfotepp	9.032	97	65			4
1,3,5-Trinitrobenzene	9.091	213	74	120		4
1-Diallate	9.118	86	43	234		4
Phenacetin	9.161	108	109	179		3
2-Diallate	9.235	86	43	234		4
Dimethoate	9.396	87	93	125		4
4-Aminobiphenyl	9.556	169	168	170		4
Pronamide	9.748	173	175	145		4
Pentachloronitrobenzene	9.748	237	295	142	214	4
Disulfoton	9.935	88	60			4
Dinoseb	9.957	211	163	147		4
thyl parathion	10.517	109	125			4
troquinoline-1-oxide	11.094	174	101	128	75	4
Parathion	11.158	109	97			4
Famphur	13.178	218	93	125		4

Methapyrilene	11.388	97	58	191		4
Aramite-1	12.435	185	191	319		5
Aramite-2	12.563	185	191	319		5
p-Dimethylaminoazobenzene	12.638	120	225	77		5
Chlorbenzilate	12.745	139	251	75		4
Kepone	17.739	272	270	237		5
3,3'-Dimethylbenzidine	13.167	212	196	106		5
2-Acetylaminofluorene	13.562	181	180	223		5
7,12-Dimethylbenz(a)anthracene	15.438	256	239	241		6
Hexachlorophene	15.747	196	198			6
3-Methylcholanthrene	16.324	268	252	253		6
INTERNAL STANDARDS						
1,4-Dichlorobenzene-d4	4.102	152	150	115		1
Naphthalene-d8	5.346	136	68			2
Acenaphthene-d10	7.601	164	162	160		3
Phenanthrene-d10	9.802	188	94	189		4
Chrysene-d12	13.926	240	236	120		5
Perylene-d12	15.902	264	265	260		6

FIGURE 1 – Tailing Factor Calculation



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$

ELEMENTS BY ICP (200.7 and 6010B)

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Approved by:

R. Wayne Potts — 24 March 2001
Date

Title: Technical Manager, QA

STL X Savannah __ Tallahassee __ Mobile __ Tampa West

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures to determine the concentration of various elements by inductively coupled plasma (ICP) atomic emission spectroscopy. This method contains the analytical procedures for the determination of metals in surface and ground water, wastewater, soil, sediment, leachate (EP or TCLP), and waste samples after digestion.

1.2 Table 1 lists the elements that may be determined by ICP and the characteristic wavelength used for each element. The reporting limit (RL) for each element, the method detection limit (MDL) for each element, and the accuracy and precision criteria for each element are in the Laboratory Quality Manual (LQM) prepared by and for STL Savannah, STL Tallahassee, STL Mobile, and STL Tampa West.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 Prior to analysis by ICP, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and exited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT). The PMTs, located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

2.2 Definitions

ICP -inductively coupled (argon) plasma; sometimes referred to a "ICAP"
TCLP-toxicity compound leaching procedure
EP (tox)-extraction procedure (toxicity)
Analytical Spike or Post-Digestion Spike - addition of a known concentration of analyte to an aliquot of sample after the preparation steps have been performed
RL - reporting limit, the lowest calibration standard or the sample equivalent of the lowest calibration standard; published in LQM or project-specific quality assurance plan (QAPP); sometimes referred to as the "practical quantitation limit(PQL).
MDL - method detection limit, the concentration that can be reported with 99% confidence that the result is greater than zero; published in LQM

2.3 This method is based on EPA Method 200.7 and SW-846 Method 6010B. Note that EPA has promulgated two versions of method 200.7-one for NPDES samples and one for drinking water. The calibration sequence for drinking water by 200.7 requires a multi-point curve with a minimum of three standards and a calibration blank.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 Each digestion lab must have acid spill kits. These kits must be located in a highly accessible area of the lab. Each digestion lab must be equipped with a properly working shower.

3.3 The standards and reagents used to prepare the standards in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.4 The Material Safety Data Sheets (MSDS) for each reagent and standard are located in each laboratory. These sheets denote the type of hazard that each reagent poses, the safe handling instructions for these compounds, and first aid instructions.

4.0 INTERFERENCES

4.1 Spectral interferences are caused by (1) the overlap of a spectral line from another element, (2) unresolved overlap of molecular band spectra, (3) background contribution from continuous phenomena, and (4) stray light from the line emissions of highly concentrated elements.

4.1.1 Spectral overlap may be compensated for by the use of inter-element correction factors.

4.1.2 Background contribution and stray light can be compensated for by a background correction adjacent to the analyte line.

4.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample digestate, by using a peristaltic pump, or by using the method of standards additions(MSA), or use of an internal standard

4.3 Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

5.1 Aqueous Samples

5.1.1 Liquid samples are routinely collected in 250-mL or 500-mL plastic containers. The sample is preserved with HNO_3 to a pH <2. The sample must be digested and analyzed within 6 months of collection. Samples may be stored at room temperature.

5.1.2 Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

5.2 Soil/Sediment Samples
Soil and sediment samples are routinely collected in 500-mL plastic containers. The sample is iced at the time of collection and is stored in the lab at 4C (less than 6C but not frozen) until time of digestion and analysis. The sample must be digested and analyzed within 6 months of collection.

5.3 TCLP or EP Toxicity Leachate Samples
The leachate is transferred to a plastic container after the extraction procedure. The sample is preserved with HNO_3 to a pH <2. The leachate sample must be digested and analyzed within 6 months of completion of the leaching procedure.

5.4 Waste Samples
Waste samples are routinely collected in 500-mL plastic containers. The sample must be digested and analyzed within 6 months of collection.

6.0 APPARATUS AND MATERIALS

6.1 Thermo Jarrell Ash TJA ICAP61E-trace, or other suitable inductively coupled plasma emission spectrometer with data system

6.2 Argon gas supply and appropriate fittings

6.3 Cooling water supply

6.4 Peristaltic pump

6.5 Volumetric flasks

6.6 Pipettes

7.0 REAGENTS
Reagents are tracked in accordance with STL SOP AN44:*Reagent Traceability.*

7.1 Reagent water-lab generated deionized water, ASTM Type I or Type II. The conductivity is monitored in accordance with STL SOP AN35.

7.2 Nitric acid (HNO_3)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals.

7.4 Hydrochloric acid (HCl)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals.

8.0 STANDARDS
Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors. Certificates of analysis or purity must be received with all neat compounds or stock solutions. All preparation steps must be in accordance with STL SOP AN41: *Standard Materials Traceability.* SOP AN43 contains guidance for the preparation of standards.

8.1 Recommended concentrations for the calibration standards are given in Table 1. Appendix A contains examples for the preparation of the initial calibration and calibration verification standards for both 6010 and 200.7. if the laboratory uses "recipes' other than those listed in Appendix A, the recipe must be documented in the standard material traceability logbook or as controlled posting. All standards must have be prepared in 5% hydrochloric acid and 1% nitric acid by volume.

NOTE: Standards must be prepared every six months "or sooner if needed or required." "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

8.2 Preparation of the Linearity Check Solutions

The linearity check solutions are prepared individually according to the following equation:

$$Vs = \frac{Vlc \otimes Clc}{Cs}$$

where
Vs = volume of stock standard (mL)
Cs = concentration of stock standard (mg/L)
Vlc = volume of linearity check standard to prepare (mL)
Clc = concentration of linearity check standard to prepare (mg/L).

The linearity check solutions are prepared at the concentrations specified in Table 1. Prepare sufficient volume to perform the linearity check, maintaining the hydrochloric acid concentration at 5% by volume and the nitric acid concentration at 1% by volume.

9.0 SAMPLE PREPARATION

The sample preparation and digestion procedures are listed in the following SOPs:

MATRIX	SOP
Aqueous and leachate samples	ME50
Soils and Sediments	ME51
Wastes and oils	ME51

10.0 ANALYSIS PROCEDURE

The analytical sequence, including standardization and calibration verification, is included in the SOP Summary in Appendix A. The SOP Summary also included the acceptance criteria for QC, including recommended corrective actions.

10.1 Initial Calibration/Standardization

10.1.1 Turn the ICP on and allow it to become thermally stable before beginning to analyze the calibration standards. It will take about an hour for the instrument to warm up. If optics were turned off, allow 2 hours warm up time.

10.1.2 Run the "Automatic Profile" program. The "automatic profile" of the instrument should be checked twice a day to compensate for changes in air pressure, humidity, and temperature. If the environment of the instrument is such that daily changes in the instrument profile are extreme, the instrument should be "profiled" every few hours.

10.1.3 Analyze the calibration standards and calibrate the ICP. ***If using a multi-point calibration, use the Calibration/Analysis and Curvefit programs to calibrate the instrument.***

10.1.4 The highest concentration calibration standard is reanalyzed after the instrument is standardized as an "unknown". The results for the re-analysis of the highest concentration calibration standard must be within +/- 5% of the true value for each target analyte. If the result for any target analyte is outside of this range, the ICP may need to be "profiled" and the standardization/calibration repeated.

10.1.5 The QC Check standards (ICV) and the Calibration Blank (ICB) are analyzed as a check on the instrument calibration.

10.1.5.1 (EPA Method 6010) The results for the target compounds in the initial calibration verification (ICV) must be within the +/- 10 % of the true value.

10.1.5.2 (EPA Method 200.7) The results for the target compounds in the initial calibration verification (ICV) must be within the +/-5.0 % of the true value. **When performing 200.7 work, note that this solution should be prepared fresh weekly.**

10.1.5.3 (EPA 6010/200.7) The results for the target compounds in the initial calibration blank (ICB) must be less than the RL.

10.1.6 The RL/PQL Check Solution is analyzed to demonstrate that the ICP is capable of detecting the target compounds at or near the reporting limit (RL). The determined concentration must within +/- 50% of the true concentration.

10.1.7 The ICP Interference Check Sample is analyzed. The concentrations of the target analytes must be within 20% of the true concentrations. Pay particular attention to false positives and false negatives for elements not present in the interference check solutions.

10.2 Continuing Calibration Verification (CCV)

10.2.1 The calibration of the ICP must be verified every 10 samples by the analysis of the analysis of the QC Check Solutions (CCV) and the Calibration Blank (CCB).

10.2.1.2 (EPA Method 6010/200.7-DW) The results for the target compounds in the continuing calibration verification (CCV) must be within the +/- 10 % of the true value.

10.2.1.2 (EPA Method 200.7-NPDES) The results for the target compounds in the continuing calibration verification (CCV) must be within the +/-5.0 % of the true value.

10.2.1.3 (EPA 6010/200.7) The results for the target compounds in the continuing calibration blank (CCB) must be less than the Reporting Limit (RL).

10.2.2 ICP Interference Check Solution and the RL check solution are analyzed at the beginning and end of each analytical sequence.

10.3 Sample Analysis

10.3.1 The samples are analyzed only after the ICB/CCB and ICV/CCV criteria are met.

10.3.2 The samples are analyzed in a sequence as follows:

INSTRUMENT WARM-UP
PROFILE
INITIAL CALIBRATION (STANDARDIZATION/CALIBRATION OF THE ICP)
REANALYSIS OF HIGH CONCENTRATION CALIBRATION STANDARD AS A SAMPLE
INITIAL CALIBRATION VERIFICATION (ICV)
INITIAL CALIBRATION BLANK (ICB)
DETECTION LIMIT CHECK SOLUTION
ICP INTERFERENCE CHECK SOLUTION A (ICSA)
ICP INTERFERENCE CHECK SOLUTION AB (ICSAB)
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB

The analytical sequence must end with the analysis of the detection limit check standard, ICSA, ICSAB, CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

10.3.3 Determine the concentration of the samples and QC items using the procedures of Section 11.

10.3.3.1 If the concentration of a sample is above the linear range of the ICP, the sample digestate must be diluted and reanalyzed.

10.3.3.2 The amount of sample digestate needed to prepare the desired dilution is determined form the following equation:

$$V_{digest} = \frac{Vf_{vol}}{DF}$$

where
Vf_{vol} = final volume of diluted sample (mL)
V_{digest} = volume of sample digestate used to make the dilution (mL)

10.3.3.3 The dilution factor is calculated as follows:

$$DF = \frac{Vf_{vol}}{V_{digest}}$$

where
Vf_{vol} = final volume of diluted sample extract (mL)
V_{digest} = volume of sample extract used to make the dilution (mL)

NOTE: The following examples are based on a final volume of 100mL. It may be more convenient to prepare dilutions at smaller final volumes.

EXAMPLE
A sample digestate is analyzed and one of the target analytes exceeds the linear range of the ICP. 1.0mL of the digestate is added to a 100mL volumetric flask and the extract brought up to volume with reagent water. What is the dilution factor?

$$DF = \frac{100mL}{1.0mL} = 100$$

Dilutions must be prepared in reagent water containing 5% hydrochloric acid and 1% nitric acid by volume.

Some samples may require multiple dilutions; that is, a dilution of a dilution will have to be made. In this case, the final dilution factor is the product of the individual dilutions.

10.4 Dilution QC Check
A dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.

10.4.1 Select a sample digestate that contains one or more target analytes at a concentrations greater than 10X the reporting limit.

10.4.2 Dilute the digestate by a factor of 5 (DF=5) and analyze the dilution using the same procedures used for the un-diluted aliquot.

10.4.3 Compare the results of the diluted and un-diluted aliquots of sample digestate.

10.4.4 If the results of the dilution are within ± 10% of the results of the undiluted sample, no matrix interference is present. If the results differ by greater than ± 10%, a matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike (see section 10.4).

If the concentration of the analyte in the sample is not at least 50 times the instrument detection limit, evaluate the post-digestion spike.

10.5 Post-digestion Spike QC Check

A post-digestion spike is performed on one sample per analytical batch to determine if matrix interferences are present. This post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration is not at least 50 times the instrument detection limit. This should be the same sample selected for dilution in 10.3, above.

10.5.1 Transfer 10mL of a digestate to a suitable vial.

10.5.2 Spike the sample with 0.10mL of ICP Matrix Spike I and 0.10mL of ICP Matrix Spike II. The theoretical concentration of the post digestion spike is the same as the LCS or MS if the volume of spiking solution is discounted.

10.5.3 Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed).

10.5.4 Calculate the percent recovery of the post digestion spike:

$$\%REC = \frac{C_{ps} - C_s}{C_2} \times 100$$

where

Cps = concentration of post digestion spike (ug/L)
Cs = concentration of un-spiked sample (ug/L)
C2 = theoretical concentration of spike (ug/L)
(See 10.2.5.2)

10.5.5 Evaluate the recovery using the following decision matrix. Limits for post digestion spikes are 75-125% recovery.

Result of Post Digestion Spikes	Action
Within 75-125% limits	None
>125% recovery	Repeat analysis. Remake spiking solutions, re-spike, and reanalyze. Reanalyze un-spiked sample
<75% recovery but >50% recovery	1) Dilute and re-spike. Elevate RL accordingly (for all associated samples). 2) Spike and evaluate all associated samples. 3) Spike and evaluate all associated samples by single point MSA 4) Qualify all associated samples
<50% recovery	Dilute digestate and repeat spike. Treat all samples associated with spike in the same manner as the spiked sample (i.e., spike or dilute samples) If recoveries are not 75-125%, analyze all associated samples by single point MSA. Note – high level of target analytes may inhibit spike recovery. Consult the supervisor in events where high levels of targets appear to be interfering

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high if corrected for spike recovery.

10.5.6 The post digestion spike and the method of standard additions must not be applied to samples analyzed at a dilution that produces a significant negative response. The analyst must use good judgement when evaluating data where the sample response is negative. Where a significant negative response is present, the digestate should be diluted and reanalyzed to determine the extent of the matrix interferences.

10.6 Single Point Method of Standard Additions

Two identical aliquots of the sample digest, Vx, are taken. One aliquot is spiked with a solution of known concentration, Cs. The second aliquot is analyzed un-spiked (the small volume of standard added to the spiked sample should be disregarded). The concentration of both aliquots are measured and the sample concentration, Cx, is calculated:

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

where

S_1 = absorbance or concentration of the spiked aliquot
S_2 = absorbance or concentration of the un-spiked aliquot
Vs = Volume of spike solution

Example:		
	Sample concentration (S_2):	523 ug/L.
	Spike solution concentration (C_s):	50,000 ug/L
	Volume of spike solution (V_s):	0.10mL
	Volume of sample aliquots (V_x):	10mL
	Spiked sample concentration (S_1):	951 ug/L

C_x = [(523)*(0.10)*(50,000)]/[(951-523)*10)] = [2,615,000]/[4280] = 611 ug/L

10.7 Determination of Linear Range of the ICP

The linear range must be determined a minimum of once per year. Divisions performing CLP analyses are required to determine and document the linear range quarterly. Documentation of the linear range study must be kept on hand and be available for inspection. A summary of the linear range study must be available to the bench analyst.

10.7.1 Profile and calibrate the ICP as described in Section 10.1

10.7.2 Prepare individual standards at concentrations that are expected to define the linear range of the instrument. Use the concentrations in Table 1 for guidance. The calibration standards and the linear range standards should be matrix matched; that is, they have the same percentage of hydrochloric and nitric acids.

10.7.3 Analyze the standards following the analytical sequence described in Section 10.3. Verify the calibration after every 10 analyses.

10.7.4 Compare the concentration of the linear range standard with its true concentration.

$$PercentDifference = \left| \frac{Ccal - Ctrue}{Ctrue} \right| \otimes 100$$

where
Ccal = concentration determined from analysis
Ctrue = true concentration of the standard

If the percent difference is less than or equal to 5%, the linear range is confirmed at that concentration. If the percent difference is greater than 5%, repeat the analysis with a lower concentration.

The linear range may be extended by analyzing higher standards and evaluating the results against the 5% difference criterion. The linear range of the ICP for an analyte is the highest standard of that analyte that meets this criterion.

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Aqueous and Leachate Samples

Aqueous samples are routinely reported in mg/L while the ICP is routinely calibrated in ug/L. If the results are reported in ug/L, the conversion factor is omitted from the calculation.

11.1.1 The concentration of the target analyte in liquid samples is calculated as follows:

$$Concentration\,(mg/L) = ug/L(from\ printout) \otimes \frac{F}{V} \otimes DF \otimes \frac{1mg}{1000ug}$$

where
F = final volume of the sample digestate (L)-usually 50mL (0.050L)
V = volume of sample digested (L)
DF = dilution factor

11.1.2 The Reporting Limit (RL) of the target analyte in liquid samples is calculated as follows:

$$Concentration(mg/L) = RLqap \otimes \frac{F}{V} \otimes DF \otimes \frac{1mg}{1000ug}$$

where
RLqap = reporting limit from STL LQM (ug/L)
F = final volume of the sample digestate (L)
V= volume of sample digested (L)
DF = dilution factor

The LQM Reporting Limits assumes:
F = 50mL, V = 50mL, and DF = 1

11.2 Soil/Solid Samples

Soils and solids are routinely reported in mg/kg while the ICP is routinely calibrated in ug/L. If the results are reported in ug/kg, the conversion factor is omitted from the calculation.

11.2.1 The concentration of the target analyte in soil and solid samples is calculated as follows:

$$Concentration(mg/kg,dw) = ug/L(from\ printout) \otimes \frac{F}{W \otimes solids} \otimes DF \otimes \frac{1mg}{1000ug}$$

where
F = final volume of the sample digestate (L)
W = volume of sample digested (kg)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)
(for example, if the percent solids is 85%, the decimal equivalent is 0.85; if the %solids is 100%, the decimal equivalent is 1.0.)

11.2.2 The Reporting Limit (RL) of the target analyte in soil/solid samples is calculated as follows:

$$Concentration(mg/kg,dw) = RL_{qap} \otimes \frac{0.0010kg}{W \otimes solids} \otimes \frac{F}{0.100L} x\ DF$$

where
RL(qap) = reporting limit from LQM
W = weight of sample digested (kg)
F = final volume of the sample digestate (L)
V = volume of sample digested (L)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)

The LQM Reporting Limits assumes F = 0.100L (100mL), DF = 1, W = 0.0010kg (1.0g), and solids = 1.0

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 STL SOP AN02: *Analytical Batching* and the SOP Summary provide guidance on evaluating QC and sample data, including recommended corrective actions.

12.2 The method detection limit (MDL) is determined annually in accordance with STL SOP CA90. The concentrations of the IDL and MDL solutions are given in Section 8 of this SOP.

12.3 Determination of the Instrument Detection Limit (IDL)

The difference between the MDL and the IDL is the *digestion step*. The MDL samples are prepared and digested prior to analysis. The IDL is defined as three times the standard deviation of seven replicate analyses analyzed over three non-consecutive days. The concentrations of the IDL and MDL solutions are given in Section 8 of this SOP. See SOP CA91 for the procedures for the determination of the IDL.

12.4 The linear range of the ICP must be determined at least annually. The procedure for the determination is given in Section 10.7 of this SOP. If any calibration regression fit, other than linear, is utilized for the calibration of the ICP (i.e., Curvilinear or Full Fit), the upper limit of the linear range is the concentration of the High Standard.

12.5 Interelement correction factors (IEC) for all elements must be determined annually. Use the manufacturer's guidance for determination of the IECs. The IECs must be verified at the beginning and end of each analytical sequence.

13.0 TROUBLESHOOTING AND PREVENTIVE MAINTENANCE

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
ICAP								
Pump Tubing	X							Change.
Nebulizer							X	Clean.
Filters			X					Inspect monthly, clean or replace as needed.
Spray Chamber							X	Clean.
Quartz Torch							X	Clean and realign.

D = daily W = Weekly M = monthly Q = Quarterly SA = semi-annually A = annually AN = as needed

14.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

Excess samples, reagents, and digests must be disposed of in accordance with SOP CA70:*Waste Management*.

15.0 REFERENCES

15.1 *Methods for Chemical Analysis of Water and Waste*; U.S EPA Office of Research and Development: Cincinnati, OHIO, March 1983.

15.2 *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986.

15.3 *Methods for the Determination of Metals in Environmental Samples*; US EPA Office of Research and Development. Washington, DC.

TABLE 1

Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Std. Conc. (mg/L)	Linear Range Std. Conc. (mg/L)*	MATRIX SPIKE CONC. (mg/L)	
						Water (mg/L)	Soil (mg/kg)
Aluminum (Al)	308.215	10	1.0/5.0	0.20	800	2.0	200
Antimony (Sb)	206.838	10	1.0/0.50	0.020	10	0.50	50
Arsenic (As)	189.042 193.696	1.0	1.0/0.50	0.010	25	2.0	200
Barium (Ba)	493.409	10	1.0/5.0	0.010	10	2.0	200
Beryllium (Be)	313.042	1.0	1.0/0.50	0.0040	10	0.050	5.0
Boron (B)	249.678	10	1.0/5.0	0.050	100	1.0	100
Cadmium (Cd)	226.502 228.802	1.0	1.0/5.0	0.0050	10	0.050	5.0
Calcium (Ca)	317.933 315.887	10	1.0/5.0	0.50	800	5.0	500
Chromium (Cr)	267.716	10	1.0/5.0	0.010	25	0.20	20
Cobalt (Co)	228.616	1.0	1.0/0.50	0.010	25	0.50	50
Copper (Cu)	324.754	10	1.0/5.0	0.020	50	0.25	25
Iron (Fe)	259.940 271.441	10	1.0/5.0	0.050	800	1.0	100
Lead (Pb)	220.353	1.0	1.0/0.50	0.0050	5	0.50	50
Magnesium (Mg)	279.079	10	1.0/5.0	0.50	1000	5.0	500

TABLE 1							
Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Std. Conc. (mg/L)	Linear Range Std. Conc. (mg/L)*	MATRIX SPIKE CONC. (mg/L)	
						Water (mg/L)	Soil (mg/kg)
Manganese (Mn)	257.610	10	1.0/5.0	0.010	50	0.50	50
Molybdenum (Mo)	202.030	1.0	1.0/0.50	0.010	50	0.50	50
Nickel (Ni)	231.604	5.0	1.0/2.5	0.040	10	0.50	50
Potassium (K)	766.491	20	10/5.0	1.0	50	5.0	500
Selenium (Se)	196.026	10	1.0/5.0	0.010	25	2.0	200
Silver (Ag)	328.068	1.0	1.0/5.0	0.010	5.0	0.050	5.0
Sodium (Na)	588.995 330.231	10	1.0/5.0	0.50	20	5.0	500
Strontium (Sr)	421.552	10	1.0/5.0	0.010	100	0.50	50
Thallium (Tl)	189.042 190.801 377.572	10	1.0/5.0	0.010	30	2.0	200
Tin (Sn)	189.989	10	1.0/5.0	0.050	50	1.0	100
Titanium (W)	334.941	10	1.0/5.0	0.010	10	1.0	100
Vanadium (V)	292.402	10	1.0/5.0	0.010	50	0.50	50
Zinc (Zn)	213.856 206.200+	5.0	1.0/2.5	0.020	20	0.50	50

*For guidance only-instrument sensitivity will vary.

APPENDIX A
SOP SUMMARY

METHOD SUMMARY - ICP ANALYSIS

HOLD/STORAGE

Container	Minimum 250mL plastic bottle with plastic or Teflon-lined lid
Preservative	HNO3 to pH <2 in the field. If dissolved metals are required, filter the samples before preservation.
Storage	Liquids preserved to pH <2 may be stored at room temperature until preparation. Solid samples must be stored at 4C (less than 6C but not frozen) until preparation.
Hold Time	Samples must be analyzed within six months from the time of collection.

SAMPLE PREPARATION

Samples should be prepared with the appropriate matrix-specific procedure.

ANALYTICAL SEQUENCE

Ignite Plasma	Follow instrument manufacturer's guidelines and allow instrument to stabilize for at least 60 minutes.
Profile Instrument	Follow manufacturer's guidelines.
Initial Calibration	Calibrate with a blank and a high standard or a blank and three standards. Verify calibration by reanalyzing highest concentration standard for each element.
Initial Calibration Verification (ICV/ICB)	Analyze an initial calibration verification solution at the beginning of the run. ICV solution must come from a source other than the calibration standard source. Analyze a calibration blank after the ICV.
Continuing Calibration Verification (CCV/CCB)	Analyze a standard with concentrations at or near mid-range levels of the calibration. The CCV should be analyzed every 10 samples and at the end of the analysis run Analyze a continuing calibration blank after every CCV.
Interference Check Solutions	At the beginning and the end of an analysis run, verify the inter-element and background corrections by analyzing the interferent check solutions (ICSA & ICSAB).
Detection limit check solution	At the beginning and the end of an analysis run and verify the accuracy at the RL by analyzing a solution at the SL RL
Serial Dilution	Perform serial dilution (1/5) on a representative sample from each batch..
Post Digestion Spike Recovery.	To check for possible matrix interference, analyze a post digestion spike on a representative sample (minimum of 1 per batch). The post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration in the sample is not at least 50 times the instrument detection limit.

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 std. and 1 blank	
Initial Calibration: Multi-point-minimum 3 stds and 1 blank	Daily	Correlation ≥0.995	Recalibrate
Highest Standard	Immediately after every calibration	Recoveries within ± 5% of expected values	New initial calibration
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	SW846 = within ±10% 200.7 = within ± 5%	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within ±10% of the true value, 200.7-NPDES - within ± 5% 200.7-Drinking Water – within ±10%	Terminate the analysis, fix the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less that the RL/CRDL	Terminate the analysis, correct the problem and reanalyze the previous 10 samples
Interference check standards (ICSA/ICSAB)	At the beginning and end of an analysis run	Determined values must be within ± 20% of the true values. Pay attention to false positives and false negatives for elements not present in the solutions.	Terminate the analysis, correct the problem, recalibrate, and reanalyze all samples since the last ICS that was in control.
Lab control sample	One per batch of twenty samples or less	6010B: STL LQM 200.7: 85-115%	Redigest and reanalyze batch
Preparation blank - SW846	One per batch of twenty samples or less	|result| <RL or result <5% of the analyte level in the sample.	Redigest and reanalyze batch
Preparation blank - 200.7	One per batch of twenty samples or less	|result| <RL or result <10% of the analyte level in the sample	Redigest and reanalyze batch
MS/MSD - SW846	One set per batch of twenty samples or less	STL LQM	Flag and report data
Serial Dilution (1/5 Dilution)	One per batch of twenty samples or less	See section 10.3.4	
Post Digestion Spike	One per batch of twenty samples or less	See section 10.4.5	
Detection Limit Check Solution	At the beginning and end of an analysis run	Recovery +/-50% of the true concentration.	Stop the analysis, fix the problem and reanalyze the affected samples.

APPENDIX B
EXAMPLES OF STANDARD PREPARATION

GENERAL INSTRUCTIONS

All calibration standards must contain 5% hydrochloric acid and 1% nitric acid by volume. The following table lists the volume of each acid needed to prepare the desired final volume of standard.

Final Volume of Standard (mL)	Volume of Hydrochloric acid (mL)	Volume of Nitric Acid (mL)
100	5.0	1.0
200	10	2.0
500	25	5.0
1000	50	10

For example, to prepare 500mL of a standard:

- Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask.
- Add 5.0mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask.
- Add the volumes of the stock standards given in the table to the volumetric flask.
- Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature.

SINGLE POINT CALIBRATION STANDARDS FOR 6010

Calibration Standard 1-Calibration Blank (ICB, CCB)

Add 500mL to 600mL of reagent water to a clean 1-L volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. Dilute to a final volume of 1.0-L with reagent water. Store the standard at room temperature. Other volumes may be prepared at the discretion of the lab. The nitric acid concentration must be 1% by volume and the hydrochloric acid concentration must be 5% by volume.

Calibration STANDARD 2

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (ml)	Conc. of Cal Std (mg/L)
Silver(Ag)	1000	0.50	500	1.0
Arsenic(As)	1000	0.50		1.0
Molybdenum(Mo)	1000	0.50		1.0
Lead(Pb)	1000	0.50		1.0
Selenium(Se)	1000	5.0		10
Antimony(Sb)	1000	0.50		1.0
Thallium(Tl)	1000	5.0		10

Calibration STANDARD 3

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Beryllium(Be)	1000	0.50	500	1.0
Barium(Ba)	1000	5.0		10
Cadmium(Cd)	1000	0.50		1.0
Cobalt(Co)	1000	0.50		1.0
Chromium(Cr)	1000	5.0		10
Copper(Cu)	1000	5.0		10
Manganese(Mn)	1000	5.0		10
Nickel(Ni)	1000	2.5		5.0
Zinc(Zn)	1000	2.5		5.0

Calibration STANDARD 4

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Aluminum(Al)	10000	0.50	500	10
Iron(Fe)	10000	0.50		10
Boron(B)	1000	5.0		10
Strontium(Sr)	1000	5.0		10
Titanium (Ti)	1000	5.0		10

Calibration STANDARD 5

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Calcium(Ca)	10000	0.50	500	10
Potassium(K)	10000	1.0		20
Magnesium(Mg)	10000	0.50		10
Sodium(Na)	10000	0.50		10
Tin(Sn)	1000	5.0		10
Vanadium(V)	1000	5.0		10

MULTI-POINT INSTRUMENT CALIBRATION-200.7

For all drinking water samples (EPA 200.7) the ICP must be calibrated with a minimum of three standards and a blank. The following standards may be used for this purpose. With the Thermo Jarrell Ash software the Calibration Analysis and Curve-fit programs must be used to be successful with the multi-point calibration of the ICP instruments.

High Standard.

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Aluminum (Al)	10000	1.0	1000	10
Antimony (Sb)	1000	1.0		1.0
Arsenic (As)	1000	1.0		1.0
Boron (B)	1000	10		10
Barium (Ba)	1000	10		10
Beryllium (Be)	1000	1.0		1.0
Cadmium (Cd)	1000	1.0		1.0
Calcium (Ca)	10000	1.0		10
Cobalt (Co)	1000	1.0		1.0
Chromium (Cr)	1000	10		10
Copper (Cu)	1000	10		10
Iron (Fe)	10000	1.0		10
Lead (Pb)	1000	1.0		10
Magnesium (Mg)	10000	1.0		10
Manganese (Mn)	1000	10		10
Molybdenum Mo)	1000	1.0		1.0
Nickel (Ni)	1000	5.0		5.0
Potassium (K)	10000	1.0		10
Selenium (Se)	1000	10		10
Silver (Ag)	1000	1.0		1.0
Sodium (Na)	10000	1.0		10
Strontium (Sr)	1000	10		10
Thallium (Tl)	1000	10		10
Tin (Sn)	1000	10		10
Titanium (Ti)	1000	10		10
Vanadium (V)	1000	10		10
Zinc (Zn)	1000	5.0		5.0

Mid-Level Standard- Prepare as the CCV is prepared.

Low-Level Standard- Prepare as the RL/PQL Check Standard.

Initial Calibration Verification (ICV) Solution

Element/Stock	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of CCV Std (mg/L)
SPEX QC19	(1)	5.0	500	(2)
SPEX QC 7	(1)	5.0		(2)
Tin(Sn)	1000	0.50		1.0
Strontium (Sr)	1000	0.50		1.0
Potassium (K)	10000	0.50		10(3)
Sodium (Na)	10000	0.45		10(3)

(1) SPEX QC19 and SPEX QC7 are solutions containing multiple elements. The concentrations are given on the certificate of analysis.

(2) The final concentrations of the various elements are the same as listed in Table 1. The SPEC QC solutions are diluted by a factor of 100 from the concentration listed on the certificate of analysis.

(3) These concentrations include the contribution from SPEX Solutions QC-7

Continuing Calibration Verification (CCV) Standard

(also used as midpoint of multi-point calibration for EPA 200.7)

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of CCV Std (mg/L)
Aluminum (Al)	10000	0.50	1000	5.0
Antimony (Sb)	1000	0.50		0.50
Arsenic (As)	1000	0.50		050
Boron (B)	1000	5.0		5.0
Barium (Ba)	1000	5.0		5.0
Beryllium (Be)	1000	0.50		0.50
Cadmium (Cd)	1000	0.50		0.50
Calcium (Ca)	10000	0.50		5.0
Cobalt (Co)	1000	0.50		0.50
Chromium (Cr)	1000	5.0		5.0
Copper (Cu)	1000	5.0		5.0
Iron (Fe)	10000	0.50		5.0
Lead (Pb)	1000	0.50		0.50
Magnesium (Mg)	10000	0.50		5.0
Manganese (Mn)	1000	5.0		5.0
Molybdenum (Mo)	1000	0.50		0.50
Nickel (Ni)	1000	2.5		2.5
Potassium (K)	10000	0.50		5.0
Selenium (Se)	1000	5.0		5.0
Silver (Ag)	1000	0.50		0.50
Sodium (Na)	10000	0.50		5.0
Strontium (Sr)	1000	5.0		5.0
Thallium (Tl)	1000	5.0		5.0
Tin (Sn)	1000	5.0		5.0
Titanium (Ti)	1000	5.0		5.0
Vanadium (V)	1000	5.0		5.0
Zinc (Zn)	1000	2.5		2.5

Reporting Limit (RL) Check Standard

(also used as low point in multi-point calibrations; e.g. EPA 200.7)

Preparation of RL/PQL Stock A-ICP

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. Of Stock Std (mg/L)
Silver (Ag)	1000	0.10	100	1.0
Arsenic (As)	1000	0.10		1.0
Cadmium (Cd)	1000	0.050		0.50
Copper (Cu)	1000	0.20		2.0
Nickel (Ni)	1000	0.40		4.0
Lead (Pb)	1000	0.050		0.50
Selenium (Se)	1000	0.10		1.0
Thallium (Tl)	1000	0.10		1.0

Preparation of RL/PQL Stock B-ICP

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. Of Stock Std (mg/L)
Aluminum (Al)	10000	0.20	100	20
Boron (B)	1000	0.50		5.0
Barium (Ba)	1000	0.10		1.0
Beryllium (Be)	1000	0.040		0.40
Calcium (Ca)	10000	0.50		50
Cobalt (Co)	1000	0.10		1.0
Chromium (Cr)	1000	0.10		1.0
Iron (Fe)	10000	0.050		5.0
Magnesium (Mg)	10000	0.50		50
Manganese (Mn)	1000	0.10		1.0
Molybdenum Mo)	1000	0.10		1.0
Sodium (Na)	10000	0.50		50
Antimony (Sb)	1000	0.20		2.0
Strontium (Sr)	1000	0.10		1.0
Tin (Sn)	1000	0.50		5.0
Titanium (Ti)	1000	0.10		1.0
Vanadium (V)	1000	0.10		1.0
Zinc (Zn)	1000	0.20		2.0

Preparation of RL/PQL Stock C-ICP

Element	Conc. of Stock Std	mL of Stock Std	Final Volume of Cal Std	Conc. Of Stock Std
Potassium(K)	10000	1.0	100	100

Preparation of the RL/PQL Check Solution-ICP

RL/PQL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A-ICP	5.0	500
Stock B-ICP	5.0	
Stock C-ICP	5.0	

ICP Interference Check Solutions

Preparation of ICP Interference Check Solution A

Element	Conc. Of Stock(mg/L)	mL of Stock Std	Final Volume(mL)	Conc. (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25		500
Magnesium (Mg)	10000	25		500
Iron (Fe)	10000	10		200

Preparation of ICP Interference Check Solution AB

Element	Conc. of Stock(mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25		500
Magnesium (Mg)	10000	25		500
Iron (Fe)	10000	10		200
Silver (Ag)	1000	0.10		0.20
Arsenic (As)	1000	0.050		0.10
Barium (Ba)	1000	0.25		0.50
Beryllium (Be)	1000	0.25		0.50
Cadmium (Cd)	1000	0.50		1.0
Cobalt (Co)	1000	0.25		0.50
Chromium (Cr)	1000	0.25		0.50
Copper (Cu)	1000	0.25		0.50
Manganese (Mn)	1000	0.25		0.50
Nickel (Ni)	1000	0.50		1.0
Lead (Pb)	1000	0.025		0.050
Antimony (Sb)	1000	0.30		0.60
Selenium (Se)	1000	0.025		0.050
Thallium (Tl)	1000	0.050		0.10
Vanadium (V)	1000	0.25		0.50
Zinc (Zn)	1000	0.50		1.0
Molybdenum (Mo)	1000	0.50		1.0
Tin (Sn)	1000	0.50		1.0

ICP Matrix Spiking Solutions

ICP Matrix Spiking Solution 1 is a solution purchased from SPEX. The certificate of analysis will list the concentrations of the analytes. Store this solution at room temperature. Prepare this solution every six months or sooner if needed or required.

Preparation of the ICP Matrix Spiking Solution 2

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Boron (B)	1000	10	100	100
Calcium (Ca)	10000	5.0		500
Magnesium (Mg)	10000	5.0		500
Molybdenum (Mo)	1000	5.0		50
Potassium (K)	10000	5.0		500
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	5.0		50
Tin (Sn)	1000	10		100
Titanium (Ti)	1000	10		100

IDL/MDL Solution

The IDL/MDL solution is used in this procedure for two purposes:

1) To determine the Instrument Detection Limit (IDL) of each target compound on a quarterly basis (SOP CA91); and
2) To determine the Method Detection Limit (MDL) of each target compound on an annual basis (SOP CA90). MDLs should be digested straight and at 1:2 dilutions.

Preparation of IDL/MDL Stock A

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Silver (Ag)	1000	0.040	100	0.40
Arsenic (As)	1000	0.20		2.0
Barium (Ba)	1000	0.020		0.20
Beryllium (Be)	100	0.050		0.050
Cadmium (Cd)	1000	0.040		0.40
Lead (Pb)	1000	0.10		1.0
Antimony (Sb)	1000	0.20		2.0
Selenium (Se)	1000	0.20		2.0
Thallium (Tl)	1000	0.20		2.0

Preparation of IDL/MDL Stock B

Element	Conc. Of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. Of Std. (mg/L)
Cobalt (Co)	1000	0.030	100	0.30
Chromium (Cr)	1000	0.10		1.0
Copper (Cu)	1000	0.10		1.0
Manganese (Mn)	1000	0.020		0.20
Molybdenum (Mo)	1000	0.040		0.40
Nickel (Ni)	1000	0.10		1.0
Tin (Sn)	1000	0.20		2.0
Vanadium (V)	1000	0.060		0.60
Zinc (Zn)	1000	0.10		1.0

Preparation of IDL/MDL Stock C

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Aluminum (Al)	10000	0.10	100	10
Calcium (Ca)	10000	0.10		10
Iron (Fe)	10000	0.10		10
Magnesium (Mg)	10000	0.10		10
Potassium (K)	10000	0.20		20
Sodium (Na)	10000	0.040		4.0

IDL/MDL Solution

Preparation of IDL/MDL Stock D

Element	Conc. Of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Boron(B)	1000	0.40	100	4.0
Strontium (Sr)	1000	0.040		0.40
Titanium (Ti)	1000	0.050		0.50
Sodium (Na)	10000	2.0		200

Preparation of the IDL/MDL Check Solution

IDL/MDL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A	5.0	1000
Stock B	5.0	
Stock C	5.0	
Stock D	5.0	

The IDL/MDL Check Solution contains the following elements at the given concentrations:

Element	Concentration(mg/L)
Be	0.00025
Ba, Mn	0.0010
Co	0.0015
Ag, Cd, Mo, Sr	0.0020
Ti	0.0025
V	0.0030
Cr, Cu, Ni, Pb, Zn	0.0050
As, Sb, Se, Sn, Tl	0.010
Na(1), B	0.020
Ca, Fe, Mg	0.050
Al, K	0.10
Na	1.0

(1) If the wavelength for sodium is 588.995, the lower concentration (0.020mg/L) is used for the IDL/MDL check solution. In this case, only stocks A,B, and C are used to make the IDL/MDL Check Solution. IDL/MDL Check Solutions for B, Sr, and Ti are prepared and evaluated separately.

MERCURY PREPARATION AND ANALYSIS

(Methods: EPA 7470A, 7471A, and 245.1)

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Technical Approval:

Andrea Teal — 01/30/04 (Date)

Title: QA Manager
STL Savannah

Safety Approval:

Ernest R. Watts — 02/03/04 (Date)

Title: EHSC
STL Savannah

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedure to determine the concentration of mercury by cold vapor atomic absorption spectrophotometry (CVAA). This method contains the manual preparation and the analytical procedures for determination of mercury in aqueous liquids (water), surface and groundwaters, soils, sediments, sludges, wastes and leachates (EP or TCLP) after digestion.

1.2 The reporting limit (RL), method detection limit (MDL), and the accuracy and precision criteria are listed in the current revision of the *Laboratory Quality Manual* (LQM) prepared by and for STL Savannah.

2.0 SUMMARY OF THE METHOD AND DEFINTIONS

2.1 This method is based on the absorption of characteristic radiation at 253.7nm by mercury vapor. After digestion, to convert all forms of mercury to the same oxidation state, the mercury ions are reduced to mercury by the addition of stannous chloride and aerated from solution after passing through a mixing coil. The mixture passes through a gas/liquid separator and through a drying tube. The vapor is passed through a flow cell positioned in the light path of an atomic absorption spectrophotometer. Mercury concentration is measured as a function of absorbance

2.2 Definitions – Refer to SOP AN99: *Definitions, Terms, and Acronyms* for a complete listing of applicable definitions.

CVAA - cold vapor atomic absorption
TCLP – toxicity characteristic leaching procedure
EP Tox - extraction procedure toxicity
Analytical Spike - addition of a known concentration of analyte to an aliquot of sample after the preparation steps have been performed; also called a post digestion spike
RL - reporting limit, the lowest calibration standard or the sample equivalent of the lowest calibration standard; published in LQM or project-specific quality assurance plan (QAPP); sometimes referred to as the "practical quantitation limit" (PQL).
MDL - method detection limit, the concentration that can be reported with 99% confidence that the result is greater than zero; published in LQM

2.3 This method is based on the guidance provided in SW-846 methods 7000A, 7470A, 7471A, and EPA method 245.1 (Drinking Water version).

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, the Waste Management SOP, and this document.

3.1 Specific Safety Concerns or Requirements

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Sulfuric acid is a strong oxidizer and is a corrosive. It will react violently when combined with organic compounds, possibly producing fire. Inhalation can cause irritation of the nose, throat, mucus membranes, and upper respiratory tract. Contact with the eyes can cause blurred vision, redness, pain, and even blindness.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

The making of aqua regia can produce toxic fumes and heat This procedure must be performed under a working fume hood.

The exhaust of the mercury analyzer must be vented or trapped so that mercury vapors do not enter the laboratory.

3 2 *Primary Materials Used*

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: **This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2ppm-TWA 4ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Permanganate	Oxidizer	5Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

4.1 Potassium permanganate is added to eliminate the possibility of interference from sulfide and certain organic compounds.

4.2 Chlorine is known to interfere with this analysis. Addition of extra potassium permanganate may be needed during the digestion of samples containing chloride. Also, the samples are not capped tightly during digestion so that excess chlorine can escape.

4.3 Contamination of the sample can occur when the preparation glassware and/or reagents contain mercury. Reagent blanks (method blanks) must be analyzed as a check on contamination due to sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

5.1 Aqueous samples and TCLP/EP-TOX Leachate

5.1.1 Liquid samples are collected in 250-mL plastic or glass containers. The samples are preserved with HNO3 to a pH<2. Samples must be digested and analyzed within 28 days of collection.

5.1.2 Samples for dissolved mercury should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered. The sample is stored at 4°C (less than 6°C, but not frozen) until filtration and preservation.

5.2 Soil/Sediment/Waste Samples

5.2.1 Soil and sediment samples are collected in 250-mL or 500-mL plastic or glass containers. The samples are iced at the time of collection and stored at 4°C (less than 6°C but not frozen) until

the time of digestion and analysis. Samples must be digested and analyzed within 28 days of collection.

6.0 APPARATUS AND MATERIALS

6.1 Leeman Hydra AA or other suitable automated mercury analyzer with data system and printer

6.2 Nitrogen or argon gas supply and appropriate fittings

6.3 Pump tubing of appropriate sizes for use on the Hydra AA

6.4 Volumetric glassware for making standards and reagents

6.5 Test tubes of the two sizes to fit the Hydra AA autosampler

6.6 Water bath or heating block capable of maintaining a temperature of 95 ± 5°C

6.7 Digestion glassware

7.0 REAGENTS

All reagents must be tracked in accordance with SOP AN44: *Reagent Traceability*.

7.1 Reagent water-lab generated deionized water, ASTM Type I or Type II. The conductivity is monitored in accordance with SOP AN35: *Conductivity Checks for Laboratory Deionized Water*.

7.2 Nitric Acid (HNO_3), concentrated-reagent grade

7.3 Hydrochloric Acid (HCl), concentrated-reagent grade

7.4 Aqua regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.5 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 50g of $KMnO_4$ in 1000mL of DI water.

7.6 Sodium chloride-hydoxylamine sulfate solution: Dissolve 120g NaCl and 120g hydroxylamine sulfate in DI water in a 1-L volumetric flask and dilute to volume.

7.7 Potassium persulfate, 5% solution (w/v): Dissolve 50g potassium persulfate in 1000mL DI water.

7.8 Rinse Water, 5% HCl-1%HNO3 - to a clean 2-L bottle add 1-L of reagent water. Carefully add 100mL of concentrated hydrochloric acid. Carefully add 20mL of concentrated nitric acid. Dilute to a final volume of 2L. Other volumes may be utilized providing the reagent proportions remain the same.

7.9 Stannous chloride ($SnCl_2.2H_2O$) - reagent grade, suitable for mercury determination

7.10 Stannous chloride ($SnCl_2.2H_2O$) solution - to a clean 2-L volumetric flask add 100g of stannous chloride. Add approximately 400mL of reagent water Carefully add 500mL of concentrated hydrochloric acid. Add a stirring bar and stir on a stir plate until the stannous chloride is dissolved Remove the stirring bar and dilute to volume with reagent water

7.11 Magnesium perchlorate ($Mg(ClO_4)_2$) - used as a drying agent in the drying tube. The magnesium perchlorate should be as coarse as possible.

7.12 Sulfuric Acid (H_2SO_4), concentrated reagent grade

8.0 STANDARDS

All standards must be tracked in accordance with SOP AN41: *Standard Material Traceability*.

8.1 Commercial stock standard, 1000mg/L.

8.2 Independent stock standard, 1000mg/L.

8.3 Calibration standards

8.3.1 Mercury calibration intermediate stock standard, 10mg/L: Add 1mL of the commercial stock standard, 1000mg/L, and 2.5mL of nitric acid to about 50mL of DI water in a 100-mL volumetric flask and dilute to volume.

8.3.2 Mercury intermediate working standard, 500µg/L: Add 5mL of the 10mg/L intermediate stock standard and 2.5mL of nitric acid to about 50mL of DI water in a 100-mL volumetric flask and dilute to volume.

8.3.3 *Mercury Calibration Standards: Transfer 0.0, 0.02, 0.04, 0.1, 0.3, and 0.5mL portions of the* intermediate working standards to a series of 125mL glass bottles. Add DI water from a graduated cylinder to each bottle to make a final volume of 50mL. This results in working standard concentrations of 0.0, 0.2, 0.4, 1.0, 3.0, and 5.0µg/L mercury. Mix well and add 2.5mL of concentrated H_2SO_4, 1.25mL of concentrated HNO_3, and 7.5mL of $KMnO_4$ solution and let stand at least 15min. Add 4mL of potassium persulfate and heat for 2 hours in a water bath at 95°C. Cool and add 3mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. The standards are ready for analysis. Larger volumes of standards may be digested as needed as long as reagent ratios are kept the same.

8.4 Calibration verification standards - Initial (ICV) and Continuing (CCV):

8.4.1 Verification intermediate stock, 5.0mg/L: Add 0.5mL of the Independent stock standard, 1000mg/L, and 2.5mL of nitric acid to about 50mL of DI water in a 100-mL volumetric flask and dilute to volume.

8.4.2 Verification Working Stock, 250µg/L: Add 5mL of the verification intermediate stock, 5.0mg/L, and 2.5mL of nitric acid to about 50mL of DI water in a 100-mL volumetric flask and dilute to volume.

8.4.3 Initial Calibration Verification (ICV) Standard, 3.0µg/L: Add 0.60mL of the verification working stock, 250µg/L, to a 125-mL glass bottle and add enough reagent water from a graduated cylinder to make a final volume of 50mL. The ICV is now ready to be digested. Other final volumes may be used as long as the reagent ratios are kept the same.

8.4.4 Continuing Calibration Verification (CCV) Standard, 2.5µg/L: Add 0.50mL of the verification working stock, 250µg/L, to a 125-mL glass bottle and add enough reagent water from a

graduated cylinder to make a final volume of 50mL. The CCV is now ready to be digested. Other final volumes may be used as long as the reagent ratios are kept the same

8.5 QC Standards

8.5.1 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Working Stock, 100µg/L: Add 2mL of the verification intermediate stock, 5.0mg/L, and 2.5mL of nitric acid to about 50mL of DI water in a 100-mL volumetric flask and dilute to volume.

8.5.2 MS/MSD, 1.0µg/L (0.05 mg/kg): To two portions of a selected sample (50mL for aqueous or 1.0-1.2g of a solid sample) add 0.5mL of the MS/MSD working standard. The MS/MSD is now ready for digestion. Where sufficient sample is available, at least 1 set of MS/MSDs should be prepared and analyzed with each batch of 20 samples or less.

8.5.3 Analytical Spike Stock Standard, 73.26µg/L: Add 2mL of the verification intermediate stock, 5.0 mg/L, and 2.5mL of nitric acid to about 50mL of DI water and dilute to 136.5mL final volume

8.5.4 Analytical Spike, 1µg/L: To a 10mL portion of digested sample, add 0.1mL of the Analytical Spike Stock Standard. One analytical spike is required per batch of twenty samples or less.

8.5.5 Lab Control Standard for Water Samples (LCSW) - 2.5µg/L: Add 0.50mL of the verification working stock, 250µg/L, to a 125mL glass bottle and add enough reagent water from a graduated cylinder to make a final volume of 50mL. The LCSW must be digested/prepared in the same manner as the samples. At least 1 LCSW must be digested with each batch of 20 samples or less.

8.5.6 Lab Control Standard for Soils Samples (LCSS): Weigh an appropriate weight of a certified solid reference standard into the digestion bottle, an example would be 0.10g of the NIST 2709 San Joquin Soil The lab control standard must be digested/prepared in the same manner as the samples.

9.0 SAMPLE PREPARATION

This section describes the manual digestion procedures for aqueous, soils/wastes, and biological matrices.

9.1 Liquid samples

9.1.1 Mix the sample thoroughly and add 50mL of sample or an aliquot of sample diluted to 50mL to a 125-mL glass bottle.

9.1.2 Add 1.25mL HNO_3, 2.5mL H_2SO_4, and 7.5mL of $KMnO_4$ solution to each sample. Shake well after each addition. Be sure the purple color of $KMnO_4$ persists for at least 15min If not, add up to three times more $KMnO_4$ solution. Equal quantities of $KMNO_4$ must be added to the LCS and MB.

9.1.3 Add 4mL of potassium persulfate to each sample, shake well, and place the samples in a water bath or block digestion apparatus at 95 ± 5°C for 2 hours.

9.1.4 Remove and allow the samples to cool. Add 3mL of hydroxylamine sulfate solution to each bottle to neutralize excess $KMnO_4$ Follow the analysis procedure below (Section 10)

9.1.5 If a different final volume is obtained (due to additional $KMNO_4$ or other reason) a dilution factor must be obtained in order to correct the final result.

9.2 Soil/Solid Samples

9.2.1 Homogenize the sample thoroughly and weigh between 1.0g and 1.2g wet weight of sample into a 125-mL glass bottle

9.2.2 Add 2.5mL DI water and 2.5mL aqua regia Heat for 2min in water bath or digestion block at 95°C.

9.2.3 Allow the samples to cool to room temperature and add 25mL DI water and 7.5mL $KMnO_4$ solution to sample. Mix and heat for 30 minutes at 95 ± 5°C.

9.2.4 Allow the samples to cool to room temperature and add 3mL sodium chloride-hydroxylamine sulfate solution to reduce excess $KMnO_4$. Add 27.5mL DI water and shake well. Follow the analysis procedure below (Section 10). If additional volume(s) of $KMNO_4$ were added, compensate for the addition(s) by adding less DI water so that the final volume will remain constant.

9.3 Fish and Crustaceans
Blanks and laboratory control standards should be treated identically. All reagents that are added to the samples should be added in the same ratios to the blanks and lab control standards. Fish are calculated on an "as is" basis.

9.3.1 Weigh between 1.0g and 1.2g of the sample and place into a 125-mL glass bottle.

9.3.2 Add 2mL H_2SO_4 and 0.5mL HNO_3 to each sample and digest in the waterbath or heating block for 30min at 80°C or until the tissue is completely dissolved.

9.3.3 After samples are cooled, add 7.5mL of $KMnO_4$ (more $KMnO_4$ may be added if required), 4mL potassium persulfate solution, 25mL DI water and put samples back into water bath for an additional 90min at 30°C.

9.3.4 Remove and cool. Add 3mL hydroxylamine sulfate solution to neutralize excess $KMnO_4$. Add 26mL DI water and shake well. If additional volume(s) of $KMNO_4$ were added, compensate for the addition(s) by added less DI water so that the final volume will remain constant.

10.0 ANALYTICAL PROCEDURE

10.1 Initial startup of the instrument

10.1.1 Before analysis begins inspect the system (pump tubes, mixing coil, gas/liquid separator) to see if any parts need to be cleaned or replaced.

10.1.2 Replace the drying tube with a freshly packed drying tube, making sure that the magnesium perchlorate is not packed too tightly. The vapors must be able to pass freely through the drying tube. Alternatively inspect the pre-made drying tube from Leeman Labs (120-00281-1) for discoloration and clean or replace as needed. Caution should be used if moisture is visible in the tubing that follows the drying tube

10.1.3 Fill the rinse tank with rinse water.

10.1.4 If the lamp is not already on and warmed up, turn on the lamp. The lamp must warm up for a minimum of 2 hours.

10.1.5 If the lamp is already on and warmed up, make sure the platens are tight and turn on the pump Allow a minimum of 20 minutes of pump time for the pump tubes to break in each day.

10.1.6 Fill the stannous chloride reagent bottle with stannous chloride solution. Switch the reagent line from rinse to the stannous chloride reagent bottle. Allow the reagent to reach the sample stream before starting an autosampler run

10.1.7 Fill the stannous chloride reagent bottle with stannous chloride solution. Switch the reagent line from rinse to the stannous chloride reagent bottle.

10.2 Autosampler setup

10 2 1 Fill the standard tubes with the appropriate standards for the protocol being followed.

10.2.2 Fill the labeled sample test tubes with the samples and calibration verification standards in the applicable order. An example order is as follows:

ICV - Initial Calibration Verification Standard
ICB - Initial Calibration Blank
Detection limit standard
9 SAMPLES
CCV - Continuing Calibration Verification Standard
CCB - Continuing Calibration Blank
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB

The preparation blank will be analyzed first The LCS will follow immediately after the preparation blank. The samples, matrix spikes, and duplicates will then follow with a maximum of 10 analyses between CCVs/CCBs. **All samples and control samples must be labeled with the corresponding batch ID.**

10.2.3 Enter the sample/QC IDs into the autosampler table giving each rack a unique name.

10.2.4 Load the rack(s) onto the autosampler.

10.3 Calibration of the mercury analyzer.

10.3.1 Call up the required protocol Open a new data folder.

10 3.2 Go to CALIBRATION, RESET, and reset the calibration for a new calibration.

10.3.3 Go to CALIBRATION, STANDARDS, and insure that calibration standards are entered at the proper concentrations.

10.3.4 Analyze the standards, beginning with standard 1 (Blank), proceeding from lowest to highest concentration.

10.3.5 When all calibration standards have been analyzed, go to CALIBRATION, LINE CALIBRATION. If calibration is within acceptable limits (correlation > 0.995) accept the linear calibration and print the calibration curve.

10.4 Sample analysis

10.4.1 Go to AUTOSAMPLER, SETUP Enter the Rack ID (s) and the cup numbers to be analyzed.

10.5 If the concentration of a sample is above the calibration range of the Hg analyzer, the sample digestate must be diluted and reanalyzed. The amount of digestate needed to prepare the desired dilution is determined from the following equation.

$$V(digest) = \frac{V(fv)}{DF}$$

where

V(digest) = volume of sample digestate used to make the dilution (mL)
V(fv) = final volume of diluted sample (mL)
DF = dilution factor

Samples should be diluted with digested blank solution

10.5.1 The dilution factor is calculated as follows:

$$DF = \frac{V(fv)}{V(digest)}$$

where

V(digest) = volume of sample digestate used to make the dilution (mL)
V(fv) = final volume of diluted sample (mL)
DF = dilution factor

10.5.2 If a sample exceeds the calibration range of the instrument by a factor of 5 or more the samples should be re-digested and reanalyzed with a smaller amount. This would be a good check for possible positive bias of the sample by incomplete digestion of organic compounds. Initial weights or volumes of <0.2g or <1.0mL should be avoided if possible so that a representative sample can be achieved. If, due to the level of mercury in the samples, greater dilutions are required, consult with your supervisor for further instructions.

Carryover from high concentration samples usually affects only the next one to two samples in the sequence. The two samples following an off-scale sample that is greater than 10µg/L must be reanalyzed to verify the presence or absence of mercury and the quantitation of mercury. It is the responsibility of the analyst to clearly demonstrate that all mercury results are accurate and free from carry-over contamination.

10.6 Post Digestion Spike (Analytical Spikes)
The post digestion spike is performed to verify that samples of similar matrix types are free from interferences from each batch spiked after the preparation/digestion.

10.6.1 Select at least one sample from within a batch for the post digestion spike

10.6.2 Add a known volume of a spiking solution to a known volume of sample digestate. It is suggested that the volume of spiking solution be 1% of the volume or less of the digestate to minimize the effects of volume on the post-digestion spike. The post spiking solution is prepared at a concentration that will yield a spike concentration at or near 2 times the RL when the sample digestate is spiked.

The following equation can be used to determine the volume of spiking solution required: where

$$V1 = \frac{C2 \otimes V2}{C1}$$

C_1 = concentration of spiking solution (mg/L)
V_1 = volume of spiking solution (mL)
C_2 = desired concentration of post digestion spike (mg/L)
V_2 = volume of sample used for post-digestion spike (mL)

10.6.3 Analyze the spiked aliquot and an un-spiked aliquot.

10.6 4 Calculate the percent recovery of the post digestion spike:

$$\%REC = \frac{C_{ps} - C_s}{C_2} \otimes 100$$

where

Cps = concentration of post digestion spike (µg/L)
Cs = concentration of un-spiked sample (µg/L)
C2 = theoretical concentration of spike (µg/L) (See 10.2.5.2)

10.6.5 Evaluate the recovery using the following decision matrix. Limits for post digestion spikes are 85-115% recovery.

Result of Post Digestion Spikes	Action
Within 85-115% limits	None
>115% recovery	Repeat analysis Remake spiking solutions, re-spike, and reanalyze. Reanalyze un-spiked sample.
<85% recovery but >50% recovery	Analyze all associated samples by single point method of standard addition and quantify by using MSA. Or qualify all associated samples on report. If sample concentration is less than the IDL, respike (to check for a spiking error), reanalyze, and re-evaluate.
<50% recovery	Dilute digestate and repeat spike. Analyze all associated samples by single point MSA.

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high.

10.6.6 The post digestion spike and the method of standard additions must not be applied to samples analyzed at a dilution that produces a significant negative absorbance. The first point in the MSA (un-spiked sample) should be greater than or equal to zero absorbance or the magnitude of the negative response should not exceed the reporting limit. Use good judgement when evaluating data where the absorbances are negative. The digestate should be diluted and reanalyzed to determine the extract of the matrix interferences.

10.7 Single Point Method of Standard Additions

Two identical aliquots of the sample digest, Vx, are taken. One aliquot is spiked with a known concentration, Cs. The second aliquot is analyzed un-spiked (the small volume of standard added to the spiked sample should be disregarded). The absorbance of both aliquots are measured and the sample concentration, Cx, is calculated:

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

where

S1 = absorbance of the spiked aliquot
S2 = absorbance of the un-spiked aliquot

10.8 Serial Dilution Check
A dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.

10.8.1 Select one sample per batch for a serial dilution analysis. Analyte concentration should be ≥25 times the instrument detection limit.

10.8 2 Dilute the digestate by a factor of 5 and analyze the dilution using the same procedure used for the unspiked aliquot.

10.8.3 Compare the results of the diluted and undiluted aliquots of sample digestate.

10.8.4 If the results of the dilution are within ±10% of the results of the undiluted sample, no matrix interference is present. If the results differ by greater than ±10% matrix interference should be suspected and the batch post-digestion spikes should be evaluated.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Aqueous and Leachate Samples

The concentration of mercury in liquid samples is routinely reported as µg/L and is calculated as follows:

$$C(sample) = C(curve) \otimes DF$$

where

C(sample) = concentration of sample (µg/L)
C(curve) = concentration from curve (µg/L)
DF = dilution factor

The RL(in µg/L) is calculated as follows:

$$RL(sample) = RL(lqm) \otimes DF$$

where

RL(sample) = reporting limit of sample (µg/L)
RL(lqm) = reporting limit from LQM (µg/L)
DF = dilution factor (The RL in the LQM assumes that DF=1)

The reporting limit (RL) may also be reported in mg/L. Results in mg/L are reported by dividing the result in µg/L by 1000.

11.2 Soil/Solid Samples

The concentration of mercury in soil and solid samples is routinely reported as mg/kg on a dry weight basis and is calculated as follows:

$$C(sample) = C(curve) \otimes \frac{F \otimes DF}{W \otimes solids} \otimes \frac{1mg}{1000ug}$$

where

C(sample) = concentration of sample (mg/kg dw)
C(curve) = concentration of digest from curve (µg/L)
F = final volume of digest (L)
W = weight of sample digested (kg)
solids = (percent solids)/100
DF = dilution factor

The reporting limit (RL) for soil/solid samples is calculated as follows:

$$RL(sample) = RL(lqm) \otimes DF \otimes \frac{1.0g}{W \otimes solids}$$

where

RL(sample) = reporting limit of sample (mg/kg dw)
RL(lqm) = reporting limit from LQM (mg/kg)
W = weight of sample digested (kg)
solids = (percent solids)/100
DF = dilution factor

RL (LQM) is based on a 1-gram sample with a percent solids of 100 (solids =1).
This equation assumes that all digests are taken to the same final volume as the standards

12.0 QUALITY CONTROL AND DATA ASSESSMENT

12.1 SOP AN02: *Analytical Batching and Evaluation of QC Data* and the SOP Summary provide guidance on evaluating QC and sample data. This guidance, including corrective actions, is summarized in Appendix A.

SOP AN02 contains the equations for the evaluation of the QC samples for accuracy and precision as well as corrective actions.

12.2 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP CA85: *Nonconformance and Corrective Action Procedures*. CA85 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures.

13.0 METHOD PERFORMANCE

The Reporting Limits (RL), the Method Detection Limits (MDL), and accuracy and precision limits associated with these methods are given in the current revision of the Laboratory Quality Manual prepared by and for STL Savannah.

13.1 Initial and Continuing Demonstration of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP *CA92: Procedure for Initial and Continuing Analyst Demonstration of Capability*.

13.2 Method Detection Limit

The method detection limit must be determined for each analyte in accordance with SOP CA90: *Procedures for the Determination of Method Detection Limit (MDL)*.

14.0 PREVENTATIVE MAINTENANCE AND TROUBLESHOOTING

14.1 Pump tubing: Inspect daily and replace as needed.

14.2 Standard Autosampler Cups: Clean daily and replace as needed.

14.3 Drying Tube: Repack daily, or more often if needed. Alternatively use the drying tube from Leeman Labs (120-00281-1) and clean and replace as needed.

14.4 Mixing Coil/Gas-Liquid Separator: Inspect weekly, clean and replace as needed.

14.5 Sample Probe: Inspect monthly, clean and replace as needed.

14.6 Mercury Lamp: Clean or replace as needed.

15.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Follow the guidance for disposal in SOP CA70: *Waste Disposal*. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment

Excess samples, reagents, and standards must be disposed in accordance with SOP CA70: *Waste Management*.

15.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

- Acidic sample digestions – Neutralize before disposal into drain/sewer system

- Excess oil samples – Transfer to waste department for storage/disposal

16.0 REFERENCES

STL Savannah's *Laboratory Quality Manual (LQM)*, current revision

Severn Trent Laboratories' *Quality Management Plan (QMP), current revision*

Test Methods for Evaluating Solid Waste, Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986 (SW-846 Update III).

Methods for Analysis of Water and Waste; U S. EPA Office of Research and Development: Cincinnati, OH, March 1983

17.0 TABLES, DIAGRAMS, AND VALIDATION DATA

Appendix A contains an SOP Summary which includes:

- Collection, preservation, and HT summary
- Analytical Sequence
- QC Criteria Summary

Appendix A - SOP SUMMARY

Collection, Preservation, and Hold Times

Container	**Aqueous:** Minimum 250mL plastic or glass bottle with a plastic or Teflon-lined lid. **Soils:** Minimum 250mL plastic or glass bottle with a plastic or Teflon-lined lid. If other metals are being tested, the aliquot form mercury may be taken from the same container
Preservation	**Aqueous:** HNO3 to pH <2 in the field If dissolved mercury is required, filter the samples before preservation. **Soils:** No chemical preservation required
Storage	**Aqueous:** Room temperature if properly preserved **Solids** should be stored at 4°C (<6°C , but not frozen) from collection until preparation.
Hold Time	**Aqueous and Soils:** Aqueous and Soils: Samples must be analyzed within 28 days of collection.

Wastes are treated in the same manner as soils.

ANALYTICAL SEQUENCE	
Instrument Startup	Turn on the mercury analyzer according to the instrument manufacturer's recommendations. Allow the mercury lamp Proper warm-up time. Inspect and change pump tubes and drying tubes as needed Check and align lamp and cell According to the instrument manufacturers recommendations.
Initial Calibration	Beginning with the blank, calibrate with the blank and 5 standards. One standard must be at or below the RL.
Initial Calibration Verification (ICV/ICB)	Analyze an initial calibration verification solution at the beginning of the analysis run. The ICV Solution must come from a source other than the calibration source. Analyze a calibration blank after the ICV.
Continuing Calibration Verification (CCV/CCB)	Analyze a standard with a concentration at or near mid-range levels of the calibration. The CCV should be analyzed every 10 samples and at the end of the analysis run. The CCV and ICV may be the same solution. Analyze a calibration blank after every CCV.
Detection Limit Check Solution	At the beginning of the analysis run, verify the accuracy at the RL by analyzing a standard with a concentration at or below the required RL.
Post Digestion Spikes/Serial dilution	At a minimum of once per analytical batch, verify the absence of matrix interference by analyzing a post digestion spike and a serial dilution.

Appendix A - SOP SUMMARY

QC CRITERIA

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 blank and 5 standards Correlation > 0.995	Recalibrate
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	SW846 = within ± 10% 245.1 = within ± 5%	Recalibrate
Continuing Calibration Verification Standards (CCV)	At the beginning and end of the analysis and every 10 samples.	SW846 = within ± 20% 245.1 = within ±10%	Terminate the analysis, correct the problem and reanalyze all samples since the last compliant CCV.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less than the required RL.	SW846 = terminate the analysis, Correct the problem and reanalyze all samples since the last compliant CCB.
RL standard (detection limit standard CRA)	After every calibration but not before the ICV.	50-150% of true value	Recalibrate.
Laboratory control sample (LCS)	One per batch of twenty or fewer samples	LQM Limits	Redigest and reanalyze batch
Preparation Blank - SW846	One per batch of twenty or fewer samples	Result < required RL.	Re-digest and reanalyze batch (if sample result >20X the blank, the sample does not have to be re-digested/reanalyzed)
MS/MSD - SW846	One set per batch of twenty or fewer samples	%Rec = 80 – 120% %RPD = < 20%	Flag and report data
MS – 245.1	MS added to a minimum of 10% of samples	%Rec = 70 - 130%	Flag and report data
Serial Dilution Analysis (1+4 dilution)	One per batch of twenty or fewer samples	If sample is at least 25 times the instrument detection limit the serial dilution, corrected for the dilution factor, should agree within +/- 10% of the undiluted sample. (Section 10.8)	Evaluate the post-digestion spike.
Post Digestion Spikes	One per batch of twenty or fewer samples	%Rec = 85 - 115% (Section 10.6)	Check for interference source and reanalyze samples, dilute all samples, or analyze all samples by MSA.

CHLORINATED HERBICIDES

(Methods: EPA 515.1, 615, and SW-846 8151A)

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Approval and Signatures

Quality Assurance Manager: Andrea Teal Date: March 21, 2005
Andrea Teal

Laboratory Director: Date: March 21, 2005
Benjamin Gulizia

EH&S Officer: Ernest B. Walton Date: March 21, 2005
Ernest Walton

SOP REVISION SUMMARY

Revision Type:

___Minor _X_ Significant ___Complete Re-write ___New SOP

Summary of Changes Since Previous Revision:

- Revised format to be consistent with current STL Savannah SOP format and NELAC requirements
- Revised safety information
- Updated the type of columns used in this procedure
- Added information on herbicide standards and correction factors to Section 8
- Revised the GC parameters in Section 10
- Removed the option to use internal standard calibration for initial calibration of the GC; not performed
- Removed information about the RL standard, no longer used
- Added quality control, method performance, preventative maintenance, and waste management information
- Revised Retention Times for target compounds listed in Appendix A
- Revised GC conditions in Appendix A
- Replaced calibration standard tables in Appendix B
- Updated the Laboratory Performance Solution Criteria in Appendix C, also included in Method Modification in Section 2

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures used to determine the concentration of chlorinated herbicides in various matrices. Appendix A contains an example of the retention time order for the herbicides, Appendix B provides examples of the calibration standards routinely analyzed, and Appendix C contains a summary of the method QC requirements for Methods 515.1, 615, and 8151A.

Method	Routine Matrices
515.1	Drinking water
615	Water and wastewater
8151	Water, groundwater, soils, solids, wastes, leachates

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits are listed in the Laboratory Quality Manual (LQM) prepared by and for STL Savannah.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 Environmental samples are prepared using the procedures outlined in SOP EX45: *Extraction of Chlorinated Herbicides.* The extracted methyl derivatives are analyzed by a GC equipped with dual capillary columns (different phases) connected to dual electron capture (EC) detectors, allowing simultaneous detection and confirmation of the target compounds. Quantitation may be performed using the external or internal standard calibration technique.

2.2 GC/MS confirmation can also be employed if analyte concentration is sufficiently high or if the sample extract is concentrated to an appropriate final volume. The esterified extract must be used for the GC/MS confirmation – do not use the 8270 extract.

2.3 Method Clarifications/Default Procedures

Elimination of Calibration Points: When more calibration standards are analyzed than required, individual compounds may be eliminated from the lowest or highest concentration level(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of a calibration be eliminated without eliminating the entire level.

Bracketing Sample Extracts: The default procedure for continuing calibration verification for the 8000-series methods is to bracket samples by CCV standards (before and after) if external standard calibration is used and ***not*** to cap the sequence (run CCV after the samples) if internal standard calibration is used ***unless*** noted in the client QAPP or in an STL pre-project plan. The internal standard provides verification information on the sensitivity and retention time stability of the instrument and verification of acceptable injections of the sample extracts. See Appendix C for summaries of the analytical sequences.

Grand Mean: The "grand mean" is used to evaluate calibration data according to the provisions of SW-846 Method 8000B and Sections 10.3 and 10.4 of this SOP.

Dilutions: *Unless otherwise specified by a client or QA plan, results from a single dilution are* reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve. For example, a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client. Project managers and lab staff must work together to balance client satisfaction with productivity.

2.4 This method is based on the guidance in SW-846 Methods 8000B, 8151A, 40 CFR 136 Method 615, and EPA method 515.1.

2.4 Method Modification:

EPA Method 515.1 requires the analysis of a Laboratory Performance Check (LPC). In addition to the sensitivity check for dinoseb, the EPA 515.1 LPC includes checks for chromatographic performance (using 4-Nitrophenol) and column performance (using 3,5-Dichlorobenzoic acid and 4-Nitrophenol). The laboratory does not report 4-Nitrophenol or 3,5-Dichlorobenzoic acid as these analytes are not regulated drinking water analytes; therefore, only the dinoseb sensitivity check is required by this SOP.

2.5 Definitions – Refer to SOP AN99: *Definitions, Terms, and Acronyms* for a complete listing of applicable definitions.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.1 Specific Safety Concerns or Requirements

Hexane is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

4.1 Glassware should be scrupulously cleaned and solvent-rinsed in accordance with SOP AN60: *Glassware Cleaning Procedures* to minimize artifacts and/or elevated baselines in gas chromatograms. Any vessel that comes in contact with the extract is a potential source for contamination. Method blanks that are extracted and analyzed with each batch of samples will provide clues to the source of contamination from the glassware and reagents.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The sample may require dilution prior to analysis to reduce or eliminate interferences. The extraction procedure SOP EX45 has several steps that are designed to eliminate or minimize interferences due to matrix. The extract is diluted as needed for data analysis. If a cleanup is used, the method blank and lab control standard must also be subjected to the cleanup.

4.3 Injection port maintenance is very important for the consistent detection of the reactive herbicides such as dinoseb.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Refer to Appendix C for a summary of the sample collection, storage, and preservation requirements.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph (GC), temperature programmable, equipped with dual electron capture (EC) detectors and a compatible autosampler

6.2 Data system compatible with the GC, with appropriate software or integration capabilities

6.3 The following column pairs are recommended. Other columns/phases may be used if the calibration and QC criteria are met and adequate separation of the target compounds is achieved.

J&W DB-XLB 30 M x 0.32 mm ID x 0.5 um film
J&W DB-35MS 30 M x 0.32 mm ID x 0.5 um film

6.4 Microsyringes: appropriate volumes

6.5 Volumetric flasks: Class A, appropriate volumes

6.6 Autosampler vials, septa, and caps: compatible with the autosampler

7.0 REAGENTS

The preparation of reagents must be performed in accordance with SOP AN44: *Reagent Traceability.*

Hexane - pesticide grade or equivalent, for preparation of standards

8.0 STANDARDS

8.1 The preparation of the calibration standards must be tracked in accordance with SOP AN41: *Standard Material Traceability.* General guidance on the preparation of standards is given in SOP AN43: *Standard Preparation.*

8.2 The lab should purchase certified solutions from STL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SOP AN43 for guidance for standard preparation.

8.3 Herbicide standards are purchased as methyl esters; therefore, the concentration of the standard must be corrected to the free acid concentration. This will eliminate the need to correct the final concentration of the sample. The correction factors are given in Appendix D.

8.4 Calibration Standard Recipes

The recipes used for standard preparation must be clearly documented as a controlled posting or as a narrative in the traceability log. The lowest level calibration standard should be at or below the equivalent of the reporting limit as defined in the LQM or client QAPP. The remaining standards will define the working range of the analytical system. Appendix B contains example recipes of the calibration levels for the routinely determined herbicides.

9.0 SAMPLE PREPARATION

The sample preparation and cleanup procedures are described in SOP EX45

10.0 ANALYTICAL PROCEDURE

10.1 Gas Chromatograph Operating Conditions

The instrument conditions listed in this section are for guidance. The actual conditions used by the lab must be documented in the instrument maintenance log, data system, or run log. The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

10.1.1 Two configurations are routinely used for the analysis of herbicides. A single column may be connected to the injection port or two columns may be connected to the injection port using a press-tight glass y-splitter and a guard column, a two-hole ferrule, or a glass tee to provide

simultaneous detection and confirmation of the target analytes.

10.1.2 Example GC Parameters

Columns:
J&W DB-XLB 30 M x 0.32 mm ID x 0.5 um film
J&W DB-35MS 30 M x 0.32 mm ID x 0.5 um film
Injector:240°C
Detector: 300°C
Carrier Gas Flow: Helium at ~2mL/min (per column) (pressure at 20psi, constant)
Make-up Gas Flow: Nitrogen at ~60mL/min (per detector)

Temperature program:

Initial Temp:	50°C
Initial Hold:	0.50 min
Program Rate 1	12°C/min to 100°C
HoldTemp 1:	15°C/min to 200°C
Program Rate 2	80°C/min
FinalTemp :	300°C (hold for 1.25 minutes)
TOTAL TIME	15.73 minutes

NOTE: These conditions and parameters are given for guidance. The columns/phases, GC conditions, and instrument parameters may be modified to optimize the analytical system.

10.2 Initial Calibration

Internal or external standard calibration techniques may be employed for the determination of the concentration of herbicides. Pentachloronitrobenzene (PCNB) may be a suitable compound to use as an internal standard

10.2.1 Prepare and analyze the calibration standards. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards. Guidance for establishing the analytical sequence is given in the SOP Summary.

Note that the following offers two options for calibration and quantitation – average CF or regression curve. Only one needs be chosen per analyte.

10.2.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the hexane blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed? Is the column properly seated in the injector and detector ports?
>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?
>Are the peaks symmetrical? Is there tailing or fronting?
>Are the areas of the peaks normal for the sensitivity setting being used?

Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

10.2.3 Evaluate the calibration curve in accordance with SOP AN67: *Evaluation of Calibration Curves.*

10.3 Initial Calibration Criteria:

Method 515.1: If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

Method 615: If the relative standard deviation is less than 10% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

Method 8151: If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

The preferred method of quantitation is the average response or calibration factor. If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve (Section 10.2.5). The "grand mean exception" described below should be applied to 8151A initial calibrations only in extraordinary circumstances because of the difficulty of maintaining and providing documentation on an on-going basis.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average calibration factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20% and no single compound has a %RSD greater than 60%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required since the lab has demonstrated that the lowest standard in the calibration curve (the equivalent of the RL) can be detected.

Regression Curve Option: A calibration curve is established for each analyte by plotting the concentration along the x-axis and the corresponding response along the y-axis. If r^2 is greater than 0.99, the curve can be used to quantify samples. For 8000-series methods, a minimum of five points is required for a linear regression, six points for a second order curve, and seven or more for higher order fits. It is recommended to use only linear and quadratic (second order) curves for quantitation. See SOP AN67 for guidance on evaluation of calibration curves.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

10.4 Calibration Verification

Calibration is verified at the frequency given in the SOP Summary. If external standard calibration is used, the following criteria apply to calibration standards analyzed before and after samples. In situations where compounds fail criteria high and no positive hits for the compound(s) failing high are detected, these samples may be reported.

If internal standard calibration is used, the samples do not have to be bracketed (capped) by the analysis of a CCV standard unless specified by a regulatory agency or client QAPP.

10.4.1 Analyze a mid-level standard. The concentration of the verification standard should be varied periodically to evaluate the calibration curve in the lower and upper halves. Tabulate the area of the target analytes and calculate the response factors if using the average RF/CF option. If using the calibration curve option, calculation of the RF is unnecessary.

Calculate the percent drift or percent difference between the initial and continuing calibration in accordance with SOP AN67.

10.4.2 Continuing Calibration Criteria

Response Criteria

If the CCV criterion is not met, another CCV should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the calibration verification criteria cannot be met, a new initial calibration must be prepared, analyzed, and evaluated.

Method 515.1: If the percent drift or percent difference is less than or equal to 20%, the initial calibration is verified and the average response factor or regression curve can be used for quantitation.

Method 615: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor or regression curve can be used for quantitation.

Method 8151: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor or regression curve can be used for quantitation.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average calibration factor or regression curve from the initial calibration can be used for quantitation if the average %drift or average % difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15% and no single compound is greater than 45%D.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

All samples analyzed using external standard calibration must be bracketed by acceptable CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

Retention Time Criteria
The retention time for the CCV must fall within the daily retention time window as defined in SOP AN66: *Determination of Retention Time Windows for Gas Chromatographic Analyses.*

Internal Standard Response Criteria
If internal standard calibration is used, the response of the internal standard(s) must be within -50% to +150% of the response in the CCV-level standard in the initial calibration sequence. If

the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

10.5 Sample Analysis Sequence

The analytical sequences for the methods are given in the SOP Summary in Appendix C. The default is to exclude QC items (method blanks, LCS, and MS/MSD) in determining the maximum number of extracts in the clock. For 8151A, more than 20 extracts (samples and QC) may be analyzed in a sequence, as long as the 12 hour time frame has not elapsed, but the number of samples (non-QC extracts) may not exceed 20. Note that some client and agency QAPPs may require that the QC items be counted as part of the twenty samples.

10.5.1 The sample extract is injected using the same injection volume used for the calibration standards. Extracts that are known to be relatively clean should be analyzed first. Extracts suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.5.2 If the concentration of target compounds exceeds the working range (defined by the highest standard in the initial calibration), the extract must be diluted in hexane and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve. If the internal standard calibration is used, the concentration of the internal standard in the diluted extract must be the same as in the calibration standards.

NOTE: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve. For example, a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client. Project managers and lab staff must work together to balance client satisfaction with productivity.

10.5.3 Occasionally, situations may arise where part of the chromatogram is obscured by large non-target peaks or matrix interferences (short, wide, peaks that are not well resolved). In these situations, it is permitted to report a lower RL for the target compounds that are not affected by the non-target or matrix interference and perform a dilution only for the target compounds that are affected. This anomalous situation must be discussed with the project manager and section supervisor prior to reporting the results and noted in the case narrative or anomaly report. Again, project managers and lab staff must work together to balance client satisfaction with productivity.

10.6 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SOP AN66: *Determination of Retention Time Windows and Evaluation of Retention Time Data Chromatographic Analyses.*

11.0 DATA ANALYSIS AND CALCULATIONS

Methyl ester herbicide standards must be corrected to the free acid concentration. This is performed by comparing the molecular weight of the methyl ester that of the acid ton determine a correction factor. Appendix D gives the molecular weights of the acids and esters. It also lists the correction factors and illustrates how to perform the acid-ester correction.

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times); the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration; and the integration of the peaks. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data. SOP AN66: *Determination of Retention Time Windows and Evaluation of Retention Time Data for Chromatographic Analyses* provides additional guidance for the evaluation of chromatographic data. This guidance is summarized in the following sections.

11.1 Compounds of Concern

Dalapon - this compound elutes very early in the run and may be subject to interference from co-eluting compounds and from artifacts from the extraction process.

MCPA and MCPP - these compounds have very low response in comparison to the other herbicides.

Dinoseb - this compound can be lost in the extraction process (hydrolysis step) but also may be lost if the injection port is not frequently and properly maintained.

11.2 Manual integrations must be documented in accordance with SOP AN65: *Manual Integrations.* Data systems should be adjusted to minimize operator intervention. All chromatographic peaks must be evaluated for overall peak shape and "reasonableness" of integration. Under no circumstances should manual integrations be used to change reasonable data system integrations in order to meet calibration or QC criteria.

11.3 The judgement and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks properly integrated?
- Are co-eluting peaks or matrix interferences present?
- Is the internal standard present at the correct retention time and response (-50% to150% of the response in the associated CCV)? Are the surrogates present at the expected RT or have they shifted?

Qualitative analysis

Identification of the surrogates and target compounds is based on retention time. The retention time (RT) windows calculated around the CCV retention times are used for the identification of the target compounds. The analyst should also note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target

compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect". This may only be performed when the RT of the internal standard and surrogates are at their respective retention times and there is little or no evidence of matrix interferences. If there is doubt as to whether the peak can be excluded or not, the default procedure will be to report the peak as the target compound unless another technique (for example, GC/MS) is used to determine that the target compound is not present.

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peak areas that exceed the established linear range of the calibration curve may result in significant retention time shifts; therefore, all peaks, which have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are handled using dilutions as detailed above (Section 10.5.2).

Evaluate the internal standard (if used) and the surrogates to check for shifts in retention times and to evaluate the surrogate recovery. The recovery criteria for surrogates are given in the LQM.

Internal Standard Criteria

The internal standard must be within the retention time window defined by the associated CCV. The response of the internal standard(s) must be within a range +/–50% of the response of the internal standard in the associated CCV.

If sample matrix interferences preclude the use of internal calibration for a sample extract, two options should be considered:

1) dilute the extract to minimize or eliminate the interference
2) use external standard calibration to quantify the target and surrogate compounds (if external standard calibration is used, all calibration requirements, including a capping standard, must be met - see Appendix C for the external standard sequence).

Surrogate Criteria

DCAA is used as the surrogate for herbicide analysis. Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Evaluate the surrogates in the same manner as the target compounds using the guidance in the table in Section 11.1.3.

NOTE: If the recovery of the surrogate(s) is above the upper control limit and no target compounds are detected in the sample, results may be reported. Refer to SOP AN02 regarding this issue.

Evaluate each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences, and the overall shapes of the peaks.

If the concentration is below the lowest calibration standard or MDL (if the sample is being evaluated for "J" results), the reporting limit (RL) for that compound is calculated (Section 11.2). The RL is calculated for all target compounds that are not detected on the primary analytical column. Peaks over-range are handled using dilutions as detailed above (10.5.2).

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected or if the concentration is within the calibration range with the %RPD >40, the analysis at a dilution is not necessary.

If the result for a target is above the reporting limit (RL) on the primary column, evaluate the confirmation column. Use the retention time window calculated using the CCV as guidance for the identification of the target compounds. Note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect".

If the target compound is detected on the confirmation column, the concentration of the target compound is calculated and compared to the result from the primary column. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

If the relative percent difference is less than or equal to 40%, the presence of the target compound is confirmed and the higher concentration is reported.

NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A larger relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. Flag the result to note the disparity (P flag) between the results. Alternatively, dilute the extract to a level that removes the interference and report the RL from this dilution.

The default guidance in this table assumes the following:
1) the retention time and response of the internal standard(s) are within acceptance criteria with little or no shift in RT
2) surrogate recovery meets the acceptance criteria and peaks fall within the middle of it's retention time window with little or no shift in RT
3) the peak identified as the target falls in the middle of the retention time window for that compound

Default Guidance for Evaluation of Surrogates and Target Compounds in Samples, LCS, and MS

PEAK INFORMATION	COLUMN 1	COLUMN 2	%RPD	REPORT
No peak present	No peak		NA	<RL
		No peak	NA	If compound is a surrogate, re-extract. If sample is LCS, re-extract.
Peak present at RT	<E	<E	<=40%	Report highest
	<E	<E	>40%	Report result most appropriate for sample matrix. Use lowest result as default. Flag with "P"
Peak present at RT	>E	<E	<=40%	Dilute extract to get both results within the calibration curve.
	<E	>E		
	>E	<E	>40%	Report lowest result and flag with "P"
	<E	>E		No dilution required.

E = highest point in curve above which results are flagged as "E". The concentration range for target compounds is RL or MDL to E. Flag results <RL but >MDL as "J". Report result less than MDL as <RL.

MS/MSD Evaluation

If the concentration of a target analyte in the un-spiked (native) sample is more than four times the theoretical concentration of the matrix spike, the recovery is not reported and the data are flagged.

11.4 Identification "Tools"

Analysis by GC/MS (scan or SIM) may be used to confirm the presence of the target compounds (see SOP SM06: *Guidelines for SIM Analysis by GC/MS.)*

11.4.1 Relative Retention Time

The retention time of a surrogate compound or internal standard provides useful information about the stability of the GC system. If the surrogate RT has not changed, it is probable that the target analytes RTs have not changed. The relative retention time can help the analyst to evaluate a peak:

$$RRT = \frac{RT_{target}}{RT_{surrogate}}$$

The relative retention time will remain fairly constant under the same GC conditions. The expected retention time of the target can be estimated from the RRT and the RT of the reference (in this case, the surrogate):

$$RT_{target} = RRT \times RT_{surrogate}$$

The analyst must be alert for the presence of matrix interferences and evaluate the data on both columns before making an identification. Another useful tool that employs a similar idea to the RRT is to "overlay" the sample chromatogram with the calibration standard. If the chromatograms are scaled the same, the overlay provides good visual cues to the identification of the target compound.

11.4.2 Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the initial identification. The analyst is cautioned to use this approach with discretion and

with consultation with the GC supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a 2-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the GC supervisor or other manager with GC experience.

12. 0 QUALITY CONTROL AND DATA ASSESSMENT

12.1 The analytical batch is discussed in SOP AN02: *Analytical Batching and Evaluation of QC Data*, and these criteria are summarized in the SOP Summary included in Appendix C. Calculation of QC data is also given in SOP AN02.

13.0 METHOD PERFORMANCE

The Reporting Limits (RL), the Method Detection Limits (MDL), and accuracy and precision limits associated with these methods are given in the current revision of the Laboratory Quality Manual prepared by and for STL Savannah.

13.1 Initial and Continuing Demonstration of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP *CA92: Procedure for Initial and Continuing Analyst Demonstration of Capability.*

13.2 Method Detection Limit

The method detection limit must be determined for each analyte in accordance with SOP CA90: *Procedures for the Determination of Method Detection Limit (MDL).*

14.0 PREVENTIVE MAINTENANCE AND TROUBLESHOOTING

Refer to SOP AN53: *Maintenance Procedures for Laboratory Instrumentation* for routine preventive maintenance and the manufacturer's guides for trouble-shooting items.

15.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Follow the guidance for disposal in SOP CA70: *Waste Disposal.* Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Hexane extracts and hexane used to rinse glassware, columns, etc. Transfer to flammable waste containers.

16.0 REFERENCES

STL Savannah's *Laboratory Quality Manual (LQM)*, current revision

Severn Trent Laboratories' *Quality Management Plan (QMP), current revision*

Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846; including Update III. U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

Code of Federal Regulations, Title 40, Part 136; U.S. Government Printing Office: Washington, DC, July 1, 1988.

METHOD 515.1: DETERMINATION OF CHLORINATED ACIDS IN WATER BY GAS CHROMATOGRAPHY WITH AN ELECTRON CAPTURE DETECTOR Revision 4.1 Edited by J.W. Munch (1995) R.C. Dressman and J.J. Lichtenberg - EPA 600/4-81-053, Revision 1.0 (1981) J.W. Hodgeson - Method 515, Revision 2.0 (1986) D. J. Munch (USEPA, Office of Water) and T. Engel (Battelle Columbus Laboratories) - National Pesticide Survey Method 3, Revision 3.0 (1987) R.L. Graves - Method 515.1, Revision 4.0 (1989) **NATIONAL EXPOSURE RESEARCH LABORATORY OFFICE OF RESEARCH AND DEVELOPMENT U.S. ENVIRONMENTAL PROTECTION AGENCY CINCINNATI, OHIO 45268**

17.0 TABLES, DIAGRAMS, AND VALIDATION DATA

APPENDIX A – Target Compounds and Retention Times

Compound	RT COL 1	RT COL 2
Dalapon	2.18	2.17
DCAA (surrogate)	10.00	10.12
Dicamba	10.07	10.26
MCPP	10.30	10.31
MCPA	10.45	10.52
Dichlorprop	10.63	10.66
Bentazon	11.42	12.18
2,4-D	10.77	10.89
Pentachlorophenol	11.18	11.15
2,4,5-TP (Silvex)	11.28	11.30
2,4,5-T	11.50	11.62
2,4-DB	11.84	11.94
Dinoseb	11.93	11.87
Picloram	12.38	12.99
DCPA	12.52	12.66

COL1 _ J&W DB-XLB
COL2 _ J&W DB-35MS

Retention Times of Target Compounds with the following conditions:

Injector: 240°C
Detector: 300°C
Carrier Gas Flow: Helium at ~5.3mL/min (pressure at 20psi, constant)
Make-up Gas Flow: Nitrogen at ~60mL/min (per detector)

Temperature program:

Initial Temp:	50°C
Initial Hold:	0.50 min
Program Rate 1	12°C/min to 100°C
Program Rate 2	15°C/min to 200°C
Program Rate 3	80°C/min
Final Temp:	300°C (hold for 1.25 minutes)
TOTAL TIME	15.73 minutes

APPENDIX B - Example Standard Preparation Recipes

Intermediate Calibration Stock Standard

Stock Standard	Concentration (ug/mL)	Volume Added (uL) to final volume of 10mL in hexane	Final Concentration (ug/mL)
Herbicide Methyl Ester Mix	See below *	250	2.5/25/500
Pentachloroanisol	100	250	2.5
DCAA Methyl Ester	100	250	2.5
Picloram Methyl Ester	100	250	2.5
DCPA	1000	50	5.0

* Methyl Esters of MCPA/MCPP at 20000 ug/mL; Dalapon at 1000 ug/mL; 2,4-D, 2,4-DB, 2,4,5-T, 2,4,5-TP, Dicamba, Dichlorprop and Dinoseb at 100 ug/mL.

Calibration Standards

	Cal. Level 1*	Cal. Level 2*	Cal. Level 3*	Cal. Level 4**	Cal. Level 5*	Cal. Level 6*	Cal. Level 7*
Intermediate Calibration Stock Standard	100 uL	200 uL	400 uL	3000 uL	800 uL	1000 uL	2000 uL
Final Concentration	0.025/0.25/5.0 ug/mL	0.050/0.50/10 ug/mL	0.10/1.0/20 ug/mL	0.15/1.5/30 ug/mL	0.20/2.0/45 ug/mL	0.25/2.5/50 ug/mL	0.50/5.0/100 ug/mL

* 10 ml final volume in Hexane
** 50 mL final volume in Hexane

APPENDIX B - Example Standard Preparation Recipes

HERBICIDE MOLECULAR WEIGHTS AND CORRECTION FACTORS

Herbicide acid	MWacid	MWester/ether	Correction factor
2,4-D	221.04	235.07	0.940
Dalapon	142.97	157.00	0.911
2,4-DB	249.09	263.12	0.947
Dicamba	221.04	235.07	0.940
Dichloroprop	235.07	249.09	0.944
Dinsoeb	240.22	254.24	0.945
MCPA	200.62	214.65	0.935
MCPP	214.65	228.67	0.939
2,4,5-TP(Silvex)	269.51	283.54	0.951
2,4,5-T	255.48	269.51	0.948
DCAA	205.04	219.07	0.936
Picloram	241.48	255.51	0.945
Pentachlorophenol	266.35	280.37	0.950

Example Calculation

$$CF(2,4-D)=\frac{Wacid}{Wester}=\frac{221.04}{235.07}=0.94$$

If the standard is expressed as mass of ester per volume, convert the concentration to he acid form by multiplying by the correction factor (CF).

APPENDIX C – Method Summary

HOLD TIMES

MATRIX (method)	Chemical Preservative/ Storage*	Routine Container	Sample Hold Time	Extract Hold Time
Drinking water (515.1)	80mg sodium thiosulfate per liter; 4°C	1-L amber x 2	14 days	28 days
Groundwater and Wastewater (615 and 8151)	None; 4°C	1-L amber x 2	7 days	40 days
Soils and wastes (8151)	None; 4°C	500-mL glass	14 days	40 days

*Storage temperature is 4°C with control criteria of less than 6°C with no frozen samples

EXTRACTION SUMMARY – detailed description in SOP EX45

Aqueous - adjust 500mL of sample to pH >12 and hydrolyze for one hour; extract with methylene chloride to remove non-targets and discard solvent; adjust aqueous phase to pH <=2 and extract with diethyl ether; concentrate, esterify and dilute to 10mL final volume with hexane

Soils - acidify 30g of sample, mix with acidified sodium sulfate; and sonicate with diethyl ether; hydrolyze with KOH at pH>12 and discard solvent; adjust aqueous phase pH to <=2 and extract with diethyl ether; concentrate, esterify, and dilute to final volume of 10mL with hexane.

ANALYSIS
Dual capillary columns with dual EC; 2-5uL injection into glass tee or y-splitter; external or internal standard calibration

SURROGATE:
DCAA - 2.0ug/L

BATCH QC
Method blank
LCS/LCSD- full target list of single peak analytes
MS/MSD- full target list of single peak analytes

Analytical Sequence

Method 515.1

Initial Calibration Standards
Initial Calibration Verification (ICV)
Laboratory Performance Solution (Daily)
Client samples analyzed until 12 hour clock expires
Calibration Verification standard – vary concentration
Client samples analyzed until 12 hour clock expires
Calibration Verification standard – vary concentration

Method 615

Initial Calibration Standards
Initial Calibration Verification (ICV)
Client samples analyzed until 24 hour clock expires
Calibration Verification standard – mid-level concentration
Client samples analyzed until 24 hour clock expires

Method 8151A

Initial Calibration Standards
Initial Calibration Verification (ICV)
20 client samples or 12 hours
Calibration Verification standard – mid-level concentration
20 client samples or 12 hours
Calibration Verification standard – mid-level concentration

The sequence continues until all samples have been analyzed or until the calibration verification fails the acceptance criteria. All sample extract analyses must be bracketed by acceptable verification standards if external standard calibration is used; if internal standard calibration is used, capping of the sequence by a CCV standard is not required unless specified in an agency or client QAPP. The default procedure is not to count the QC items in the 20 sample extracts that may be analyzed in the clock; i.e.; the number of sample and QC extracts may exceed 20 but the total number of sample extracts may not exceed 20 and all extracts (samples and QC) must be analyzed within the 12-hour clock.

Laboratory Performance Solution Criteria (EPA 515.1)

Test	Analyte(s)	Concentration (ug/mL)	Criteria
Sensitivity	Dinoseb	0.004	S/N >3

S/N = a ratio of peak signal to baseline noise.

EPA Method 515.1 requires the analysis of a Laboratory Performance Check (LPC). In addition to the sensitivity check for dinoseb, the EPA 515.1 LPC includes checks for chromatographic performance (using 4-Nitrophenol) and column performance (using 3,5-Dichlorobenzoic acid and 4-Nitrophenol). Since the laboratory does not report these analytes, only the dinoseb sensitivity check is required by this SOP.

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration- 5-point minimum with lowest point at RL	Prior to sample analysis or when CCV fails	1) 515.1 %RSD of each target <=20% or r^2 >0.99 2) 615 %RSD of each target <=10% or r^2 >0.99 3) 8151 %RSD of each target <=20% or r^2 >0.99 (see Section 10.2 for 8000-series "grand mean" exception)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and analyze standards
Initial calibration verification (Second Source ICV)	After Initial Calibration	515.1: Percent difference <=20% 615/8151: Percent difference <= 15%	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Laboratory Performance Check Solution	Daily, prior to sample analyses	See criteria in previous section.	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification (CCV)	515.1/8151: After every twenty sample analyses (or 12 hours) and at the end of the sequence 615: After every twenty sample analyses (or 24 hours)	515.1:Percent difference or drift <= 20% 615/8151: Percent difference or drift <= 15% (see Section 10.3 for 8000-series"grand mean" exception)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Method blank	Per batch	All targets reported less than RL in LQM	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze -Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze
Lab control sample (LCS)	Per batch	Recoveries within LQM limits	-Evaluate chromatogram and integrations. -Check calculations and reanalyze -Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix spike (MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. - Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze
Surrogate	All samples, method blanks, and QC	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze
Initial Demonstration of Capability (IDOC)	Initially and when new analysts trained	Evaluate in accordance with SOP 92 and method criteria	Repeat test for analytes that fail criteria
Method Detection Limit (MDL)	See SOP CA90	Evaluate in accordance with SOP CA90	Evaluate in accordance with SOP CA90
Retention time window determination	See SOP AN66	See SOP AN66	See SOP AN66

ORGANOCHLORINE PESTICIDES AND PCBs BY GC

(Methods: EPA 608, 8081A, and 8082)

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Technical Approval:

Andrea Sekol — 03/22/04 (Date)

Title: Quality Assurance Manager
STL Savannah

Safety Approval:

Ernest B. Wolfe III — 03/22/04 (Date)

Title: EHSC
STL Savannah

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures used to determine the concentration of chlorinated pesticides and polychlorinated biphenyls (PCBs) as Aroclors in various matrices. Appendix A contains an example of the retention time order for the single peak pesticides, Appendix B provides examples of the calibration standards routinely analyzed, and Appendix C contains a summary of the method QC requirements for Methods 608, 8081A, and 8082.

1.2 The routine target compounds, reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria are listed in the current revision of the Laboratory Quality Manual (LQM) prepared by and for STL Savannah.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 Environmental samples are prepared using matrix-specific procedures (see Section 9). The solvent is evaporated, the residue exchanged into hexane, and the sample adjusted to a final volume of 10mL or less. The preparation may also incorporate Florisil, copper (sulfur), acid (PCBs only), or gel permeation chromatography (GPC) cleanups. Analysis of the extract is routinely performed on a GC equipped with dual capillary columns (different phases) connected to dual electron capture (EC) detectors, allowing simultaneous detection and confirmation of the target compounds. GC/MS confirmation can also be employed if analyte concentration is sufficiently high or if the sample extract is concentrated to an appropriate final volume. Quantitation may be performed using the external or internal standards calibration technique.

2.2 Method Clarifications / Default Procedures

General Clarification: The procedures for chlorinated pesticides (8081A) and PCBs (8082) are given as separate methods in Update III of SW-846. In previous updates, pesticides and PCBs were included in a single method; pesticides and PCBs are still included in the scope of EPA Method 608. The extraction and the analysis are combined in this SOP 1) to reduce the time of extraction and analysis; and 2) to reduce the amount of solvent used in the procedures (one extraction instead of two). If interferences or high levels of non-PCB compounds are present, a portion of the extract can be subjected to the acid cleanup and reanalyzed. Note that if the list of target analytes includes only a limited list of components (i.e. Toxaphene, Chlordane, or PCBs), these procedures may be abbreviated to address only the analytes of interest.

Bracketing Sample Extracts: The laboratory's default procedure for continuing calibration verification stems from EPA Method 8000 and is to bracket samples by CCV standards (before and after) if external standard calibration is used and ***not*** to cap the sequence (run CCV after the samples) if internal standard calibration is used ***unless*** noted in an agency or client QAPP, in an STL pre-project plan, or in the method. EPA method 8081 specifically states to perform bracketing CCV every 12 hours (or 20 samples) and at the end of the analytical sequence; therefore, the requirement for capping CCV has been incorporated into this SOP.

Grand Mean: The "grand mean" is used to evaluate calibration data according to the provisions of SW-846 Method 8000B and Sections 10.3 and 10.4 of this SOP.

Quantitation of QC Items: The default procedure for the analysis and evaluation of QC items (method blank, LCS, and MS/MSD) is to analyze these items on one of the instruments used to analyze the associated samples.

Dilutions: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the

upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve. For example, a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client. Project managers and lab staff must work together to balance client satisfaction with productivity.

2.3 This method is based on the guidance in SW-846 Methods 8000B, 8081A, and 8082, and 40 CFR 136 Method 608.

2.4 Definitions – Refer to SOP AN99: *Definitions, Terms, and Acronyms* for a complete listing of applicable definitions.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.1 Specific Safety Concerns or Requirements

Acetone and hexane are flammable solvents. They can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

4.1 Glassware should be thoroughly cleaned and solvent-rinsed in accordance with SOP AN60: *Glassware Cleaning Procedures* to minimize artifacts and/or elevated baselines in gas chromatograms. Any vessel that comes in contact with the extract is a potential source for contamination. Method blanks that are extracted and analyzed with each batch of samples will provide clues to the source of contamination from the glassware and reagents.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. See Section 9 for a table summary of the cleanups that may be employed to eliminate or reduce interferences. If matrix interferences continue after a cleanup has been performed, the sample is diluted as needed for data analysis. If a cleanup is used, the method blank and laboratory control standard must also be subjected to the cleanup.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Aqueous samples are collected in 1-L glass containers with Teflon-lined caps. Soil/sediment samples are collected in wide mouth glass jars equipped with Teflon lined caps. No preservative is added. The samples are iced at the time of collection and maintained at 4°C (less than 6°C with no frozen samples) until extraction. Extraction must be performed within 7 days for aqueous samples and within 14 days of sampling for soils/solids. The extracts must be stored at 4°C (less than 6°C) and must be analyzed within 40 days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph (GC), temperature programmable, equipped with single or dual electron capture (EC) detectors and a compatible autosampler

6.2 Data system compatible with the GC, with appropriate software or integration capabilities

6.3 The following column pairs are recommended. Other columns/phases may be used if the calibration and QC criteria are met and adequate separation of the target compounds is achieved.

CLP I fused silica capillary column 30 M x 0.53 mm ID x 1.5 µm film
CLP II fused silica capillary column 30 M x 0.53 mm ID x 0.83 µm film

6.4 Microsyringes, appropriate volumes

6.5 Volumetric flasks, Class A, appropriate volumes

6.6 Autosampler vials, septa, and caps - compatible with the autosampler

7.0 REAGENTS

All reagents must be tracked in accordance with SOP AN44: *Reagent Traceability.*

Hexane - pesticide grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SOP AN41: *Standard Material Traceability*. General guidance on the preparation of standards is given in SOP AN43: *Standard Preparation.*

The lab should purchase certified solutions from STL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SOP AN43 for guidance for standard preparation.

Calibration Standard Recipes

The recipes used for standard preparation must be clearly documented as a controlled posting or as a narrative in the traceability log. The lowest level calibration standard should be at or below the equivalent of the reporting limit as defined in the LQM or client QAPP. The remaining standards will define the working range of the analytical system. Appendix B contains example recipes of the calibration levels for the routinely determined single peak pesticides, technical chlordane, toxaphene, and the Aroclors.

If internal standard calibration is used, each calibration standard must contain the same concentration of the internal standard(s). The recommended concentration range for the internal standard(s) is 0.050 to 0.10µg/mL.

9.0 SAMPLE PREPARATION

The sample preparation and cleanup procedures are described in the following SOPs:

PROCEDURE	MATRIX	STL SOP
Continuous Liquid-Liquid extraction	aqueous and leachates	EX30
Ultrasonic extraction	soils and sediments	EX40
Waste dilution	Waste samples (oils, products, etc)	EX42
Zymark extract concentration	All extracts	EX50

CLEAN-UP PROCEDURE	STL SOP	APPLICATION	EFFECTIVENESS
Florisil	EX62	Pest/PCBs	Eliminates polar non-target compounds
Sulfuric acid	EX60	PCBs	Eliminates some unsaturated hydrocarbon interferences
Copper	EX60	Pest/PCBs	Eliminates elemental sulfur
GPC	EX61	Pest/PCBs	Eliminates high molecular weight non-target compounds and sulfur

10.0 ANALYTICAL PROCEDURE

10.1 Gas Chromatograph Operating Conditions

The instrument conditions listed in this section are for guidance. The actual conditions used by the lab must be documented in the instrument maintenance log, data system, or run log. The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

10.1.1 Two configurations are routinely used for the analysis of pesticides and PCBs. A single column may be connected to the injection port or two columns may be connected to the injection port using a press-tight glass y-splitter and a guard column, a two-hole ferrule, or a glass tee to provide simultaneous detection and confirmation of the target analytes.

10.1.2 Example GC Parameters

Injector: 220 – 240°C
Detector: 300 – 320°C
Carrier Gas Flow: Helium at 5 mL/min (per column)
Make-up Gas Flow: Nitrogen at 25 mL/min (per detector)-see manufacturer's recommended flows

Example chromatogram temperature program:

Initial Temp:	160°C
Initial Hold:	4.0 min
Program Rate:	10°C/min
Final Temp:	270°C (hold for 10 minutes)
Injected Volume:	2-4µL - 1-2µL per column (single injection into guard column and "Y" splitter)

NOTE: These conditions and parameters are given for guidance. The columns/phases, GC conditions, and instrument parameters may be modified to optimize each analytical system.

10.2 Column Evaluation (608 and 8081A)

10.2.1 PEVAL Breakdown Standard

The column(s) must be evaluated prior to the analysis of the calibration standards and once every 12 hour clock. (NOTE: For EPA 608, this column evaluation is required once every 24 clock.) The column evaluation is performed by injecting a PEVAL standard that contains Endrin and p,p'-DDT and calculating the percent breakdown of these compounds. The standard used for determining the percent breakdown must not contain any compounds that coelute with Endrin, DDT, or any of the corresponding breakdown products.

NOTE: This column evaluation does not have to be performed if PCBs only are the target compounds. PCBs are stable and not subject to breakdown in the injection port.

Inject the Endrin/DDT breakdown standard. Check the peak integrations and calculate the breakdown as follows:

$$\%\text{Breakdown Endrin} = \frac{\text{Response(Endrin Aldehyde + Endrin Ketone)}}{\text{Response(Endrin + Endrin aldehyde + Endrin Ketone)}} \otimes 100$$

$$\%\text{Breakdown DDT} = \frac{\text{Response(DDE + DDD)}}{\text{Response(DDT + DDE + DDD)}} \otimes 100$$

The response (area or height) must be used to evaluate the breakdown. Do not use concentrations and do not "undetect" peaks that are below the RL or MDL. All peaks detected by the data system must be included in the percent breakdown calculation.

Breakdown Criterion
The breakdown for each compound must be less than 15%. If the breakdown exceeds 15%, the instrument will require column and/or injector port maintenance. The maintenance may include but is not limited to replacing the septum, clipping the front of the guard column, replacing the glass injector sleeve, and scrubbing (cleaning) the injector port.

10.2.2 If the instrument has not been in use for more than one day, a "priming" analysis may be beneficial. The analysis of a relatively high concentration pesticide or PCB standard may help to stabilize the response of the very sensitive EC detector. Inject a standard that is about 10x the concentration of the highest calibration standard and allow the instrument to cycle through the temperature program. It is not necessary to acquire the data but the baseline should be monitored before and after the priming analysis to gauge the condition of the detector. A hexane blank should be analyzed after the analysis of the priming standard and before the % breakdown check.

NOTE: The "priming" standard should be injected manually to avoid contaminating the autosampler syringe.

10.3 Initial Calibration

Initital calibration must be performed in accordance with SOP AN67: *Evaluation of Calibration Curves*. Internal or external standard calibration techniques may be employed for the determination of the concentration of pesticides and PCBs. Pentachloronitrobenzene (PCNB) or 2-Nitro-1-bromobenzene is recommended for use as internal standards; however, other compounds may be used.

Internal standard calibration should be used as the default. If matrix interferences preclude the use of internal calibration for a sample extract, two options should be considered:

1) dilute the extract or perform sample extract cleanup to minimize or eliminate the interference
2) use external standard calibration to quantify the target and surrogate compounds (if external standard calibration is used, all calibration requirements, including a capping standard, must be met - see Appendix C for the external standard sequence).

10.3.1 Prepare and analyze the calibration standards. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards. Guidance for establishing the analytical sequence is given in the SOP Summary.

Note that the following offers two options for calibration and quantitation – average CF or regression curve. Only one needs be chosen per analyte.

10.3.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the hexane blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed? Is the column properly seated in the injector and detector ports?
>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?
>Are the peaks symmetrical? Is there tailing or fronting?
>Are the areas of the peaks normal for the sensitivity setting being used?

Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

10.3.3 Evaluate the calibration curve in accordance with SOP AN67: *Evaluation of Calibration Curves*.

10.3.4 Initial Calibration Criteria:

600-series: If the relative standard deviation is less than 10% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

8000-series: If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

The preferred method of quantitation is the average response or calibration factor. If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve. The "grand mean exception" described below should be applied to 8081A and 8082 initial calibrations only in extraordinary circumstances because of the difficulty of maintaining and providing documentation on an on-going basis.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average calibration factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%, with no individual compound exceeding 3X the ICAL criteria (60%).
NOTE: If a target compound that passes by the "grand mean exception" is detected the PM is notified via an anomaly report or case narrative.

Regression Curve Option: A calibration curve is established for each analyte by plotting the concentration along the x-axis and the corresponding response along the y-axis. If the regression coefficient of the regression curve is greater than 0.99, the curve can be used to quantify

samples. For 8000-series methods, a minimum of five points is required for a linear regression, six points for a second order (quadratic) curve, and seven or more for higher order fits. It is recommended to use only linear and quadratic (second order) curves for quantitation. See SOP AN67 for guidance on evaluation of calibration curves.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

10.4 Calibration Verification

A second source initial calibration verification standard must be performed in accordance with SOP AN67.

Calibration is verified at the frequency given in the SOP Summary. The following criteria apply to calibration standards analyzed before and after samples. In situations where compounds fail criteria high and no positive hits for the compound(s) failing high are detected, these samples may be reported if appropriately qualified.

Analyze a mid-level standard. The concentration of the verification standard should be varied quarterly to evaluate the calibration curve in the lower and upper halves. Tabulate the area of the target analytes and calculate the response factors if using the average RF/CF option. If using the calibration curve option, calculation of the RF is unnecessary.

Calculate the percent drift or percent difference between the initial and continuing calibration in accordance with SOP AN67.

10.4.2 Continuing Calibration Verification Criteria

Response Criteria
If the CCV criterion is not met, another CCV should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the calibration verification criteria cannot be met, a new initial calibration must be prepared, analyzed, and evaluated.

600-series: If the percent drift or percent difference is less than or equal to 15%, the initial calibration is verified and the average response factor or regression curve can be used for quantitation.

8000-series: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor or regression curve can be used for quantitation.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average calibration factor or regression curve from the initial calibration can be used for quantitation if the average %drift or average % difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15% and no single compounds exceeds 3X (45%) the CCV criteria.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative.

All samples must be bracketed by acceptable CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no

target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (per SW-846 Method 8000B).

Retention Time Criteria
The retention time for the CCV must fall within the daily retention time window as defined in SOP AN66: *Determination of Retention Time Windows for Gas Chromatographic Analyses.*

Internal Standard Response Criteria
If internal standard calibration is used, the response of the internal standard(s) must be within –50% to +150% of the response in the CCV-level standard in the initial calibration sequence. If the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

10.5 Sample Analysis Sequence
The analytical sequences for the 600- and 8000-series methods are given in the SOP Summary in Appendix C. The default is to exclude QC items (method blanks, LCS, and MS/MSD) in determining the maximum number of extracts in the clock.

For 8081 and 8082, more than 20 extracts (samples and QC) may be analyzed in a sequence, as long as the 12 hour time frame has not elapsed, but the number of samples (non-QC extracts) may not exceed 20. Note that some client and agency QAPPs may require that the QC items be counted as part of the twenty samples.

For 608, more than 20 extracts (samples and QC) may be analyzed in a sequence, as long as the 24 hour time frame has not elapsed, but the number of samples (non-QC extracts) may not exceed 20. Note that some client and agency QAPPs may require that the QC items be counted as part of the twenty samples.

10.5.1 The sample extract is injected using the same injection volume used for the calibration standards. Extracts that are known to be relatively clean should be analyzed first. Extracts suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

If the internal standard calibration is used, the concentration(s) of the internal standard(s) must be the same in all calibration samples, field samples, and QC samples. A concentration of 0.050ug/mL to 0.10ug/mL (final extract concentration) is recommended.

10.5.2 If the concentration of target compounds exceeds the working range (defined by the highest standard in the initial calibration), the extract must be diluted in hexane and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve. If the internal standard calibration is used, the concentration of the internal standard in the diluted extract must be the same as in the calibration standards.

NOTE: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve. For example, a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to

provide lower detection limits to the client. Project managers and lab staff must work together to balance client satisfaction with productivity.

10.5.3 Occasionally, situations may arise where part of the chromatogram is obscured by large non-target peaks (such as phthalate esters, which elute in the same general retention time range as the pesticides and PCBs) or matrix interferences (short, wide, peaks that are not well resolved). In these situations, it is permitted to report a lower RL for the target compounds that are not affected by the non-target or matrix interference and perform a dilution only for the target compounds that are affected. This anomalous situation must be discussed with the project manager and section supervisor prior to reporting the results and noted in the case narrative or anomaly report. Again, project managers and lab staff must work together to balance client satisfaction with productivity.

10.6 *Determination of Retention Time Windows*

The procedure for the determination of retention time windows is given in SOP AN66: *Determination of Retention Time Windows and Evaluation of Retention Time Data for Chromatographic Analyses.* If internal standard calibration is used relative retention times, as described in Section 11.1.4, are used to identify the target compounds.

11.0 DATA ANALYSIS AND CALCULATIONS

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times); the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration; and the integration of the peaks. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data. SOP AN66: *Determination of Retention Time Windows and Evaluation of Retention Time Data for Chromatographic Analyses* provides additional guidance for the evaluation of chromatographic data. This guidance is summarized in the following sections.

Manual integrations must be documented in accordance with SOP AN65: *Manual Integrations.* Data systems should be adjusted to minimize operator intervention. All chromatographic peaks must be evaluated for overall peak shape and "reasonableness" of integration. Under no circumstances should manual integrations be used to change reasonable data system integrations in order to meet calibration or QC criteria.

The judgement and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks properly integrated?
- Are co-eluting peaks or matrix interferences present?
- Is the internal standard present at the correct retention time and response (-50% to150% of the response in the associated CCV)? Are the surrogates present at the expected RT or have they shifted?

11.1 Qualitative analysis

Identification of the surrogates and target compounds is based on retention time. The retention time (RT) windows calculated around the CCV retention times are used for the identification of the target compounds. The analyst should also note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect". This may only be done when the RT of the internal standard and surrogates are at their respective retention times and there is little or no evidence of matrix interferences. If there is doubt as to whether the peak can be excluded or not, the default procedure will be to report the peak as the target compound unless another technique (for example, GC/MS) is used to determine that the target compound is not present.

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peak areas that exceed the established linear range of the calibration curve may result in significant retention time shifts; therefore, all peaks, which have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are handled using dilutions as detailed previously.

11.1.1 Evaluate the internal standard (if used) and the surrogates to check for shifts in retention times and to evaluate the surrogate recovery. The recovery criteria for surrogates are given in the LQM.

Internal Standard Criteria
The internal standard must be within the retention time window defined by the associated CCV. The response of the internal standard(s) must be within a range ±50% of the response of the internal standard in the associated CCV.

If sample matrix interferences preclude the use of internal calibration for a sample extract, two options should be considered:

1) dilute the extract or perform sample extract cleanup to minimize or eliminate the interference
2) use external standard calibration to quantify the target and surrogate compounds (if external standard calibration is used, all calibration requirements, including a capping standard, must be met - see Appendix C for the external standard sequence).

Surrogate Criteria
A minimum of two surrogates is spiked into each sample and QC item prior to preparation. Decachlorobiphenyl (DCB) and 2,3,4,6-tetrachloro-m-xylene (TCMX) are the recommended surrogates. DCB should be evaluated as the primary surrogate; TCMX is evaluated if there is matrix interference with DCB.

NOTE: TCMX (or an alternate surrogate such as octachloronaphthalene) is to be used to evaluate data when AR1268 is detected. That is, TCMX or the alternate surrogate must pass the surrogate recovery criterion in order to report the data without qualification. AR1268 contains DCB and will bias the recovery when DCB is evaluated as a surrogate.

Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Evaluate the surrogates in the same manner as the target compounds using the guidance in the table in Section 11.1.3.

NOTE: If the recovery of the surrogate(s) is above the upper control limit and no target compounds are detected in the sample, results may be reported with appropriate qualification. Refer to SOP AN02: *Analytical Batching and Evaluation of QC Data* regarding this issue.

Dilutions and Surrogate Recovery
The concentration range for surrogates is approximately 0.0025µg/mL to 0.080µg/mL. This should give up to a six-fold dilution to report surrogate recoveries, if the spiking level is 0.5µg/L and the lower limit of quantitation is 0.025µg/L. The lower recovery limit is 30%; therefore, the lowest acceptable concentration is 0.5*0.3=0.15µg/L. The highest dilution to report un-qualified results ("J" flag) would be 0.15µg/L/0.025µg/L = 6 or a six fold dilution.

11.1.2 Evaluate each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences, and the overall shapes of the peaks.

If the concentration is below the reporting limit standard or MDL (if the sample is being evaluated for "J" results), the reporting limit (RL) for that compound is calculated (Section 11.2). The RL is calculated for all target compounds that are not detected on the primary analytical column. Peaks over-range are handled using dilutions as detailed above (10.5.2).

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected on the confirmation column or if the concentration is within the calibration range with the %RPD >40, the analysis at a dilution is not necessary.

11.1.3 If the result for a target is above the reporting limit (RL) on the primary column, evaluate the confirmation column. Use the retention time window calculated using the CCV as guidance for the identification of the target compounds. Note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect".

If the target compound is detected on the confirmation column, the concentration of the target compound is calculated and compared to the result from the primary column. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

If the relative percent difference is less than or equal to 40%, the presence of the target compound is confirmed and the higher concentration is reported.

NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A larger relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, the analyte is confirmed; however, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. Report the result that is most reasonable for the sample. The lower result is reported as the default when the %RPD is >40. Flag the result to note the disparity (P flag) between the columns.

NOTE: The default guidance instructs the analyst to report the highest result when the %RPD <=40% and to report the lowest result when the %RPD exceeds 40%. The guidance uses RPD to determine if matrix interference is present. If the RPD is <=40%, the results between the two columns are essentially the same, and no matrix interference is deemed present. If the RPD exceeds 40%, a matrix interference is most likely present, and the lower result is reported as the default. This guidance does not prohibit reporting the higher result when the %RPD exceeds 40; however, this guidance is intended to preclude reporting clearly unreasonable data. It is this laboratory's experience that a majority of matrix interferences are positive and that reporting the lower of the two results is reasonable when the %RPD exceeds 40.

Alternatively, perform additional extract cleanup (sulfur, florisil, etc.) or if cleanup is not feasible or there is a low probability that cleanup will help, dilute the extract to a level that removes the interference and report the RL from this dilution.

The table below gives default guidance for evaluating hits.

The default guidance in this table assumes the following:
1) the retention time and response of the internal standard(s) are within acceptance criteria with little or no shift in RT
2) surrogate recoveries meet the acceptance criteria and peaks fall within the middle of the retention time window with little or no shift in RT
3) the peak identified as the target falls in the middle of the retention time window for that compound

Default Guidance for Evaluation of Surrogates and Target Compounds in Samples, LCS, and MS

PEAK INFORMATION	COLUMN 1	COLUMN 2	%RPD	REPORT
No peak present	No peak		NA	<RL
		No peak	NA	If compound is a surrogate, re-extract. If sample is LCS, re-extract.
Peak present at RT	<E	<E	<=40%	Report highest
	<E	<E	>40%	Report result most appropriate for sample matrix. Use lowest result as default. Flag with "P"
Peak present at RT	>E	<E	<=40%	Dilute extract to get both results within the calibration curve.
	<E	>E		
	>E	<E	>40%	Report lowest result and flag with "P" No dilution required.
	<E	>E		

E = highest point in curve above which results are flagged as "E". The concentration range for target compounds is RL or MDL to E. Flag results <RL but >MDL as "J". Report results less than MDL as <RL.

MS/MSD Evaluation
If the concentration of a target analyte in the un-spiked (native) sample is more than four times the theoretical concentration of the matrix spike, the recovery is not reported and the data is flagged.

11.1.5 Identification "Tools"
Analysis by GC/MS (scan or SIM) may be used to confirm the presence of the target compounds in accordance with SOP SM06: *Guidelines for SIM Analysis by GC/MS.*

11.1.5.1 Relative Retention Time

The retention time of a surrogate compound or internal standard provides useful information about the stability of the GC system. If the surrogate RT has not changed, it is probable that the target analytes RTs have not changed. The relative retention time can help the analyst to evaluate a peak:

$$RRT = \frac{RT_{target}}{RT_{surrogate}}$$

The relative retention time will remain fairly constant under the same GC conditions. The expected retention time of the target can be estimated from the RRT and the RT of the reference (in this case, the surrogate):

$$RT_{target} = RRT \times RT_{surrogate}$$

The analyst must be alert for the presence of matrix interferences and evaluate the data on both columns before making an identification. Another useful tool that employs a similar idea to the RRT is to "overlay" the sample chromatogram with the calibration standard. If the chromatograms are scaled the same, the overlay provides good visual cues to the identification of the target compound.

11.1.5.2Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the initial identification. The analyst is cautioned to use this approach with discretion and with consultation with the GC supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a 2-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the GC supervisor or other manager with GC experience.

11.1.6 Qualitative Analysis of Multiple Peak Compounds

Identification of multi-peak pesticides (Toxaphene and Technical Chlordane) and PCBs as Aroclors is based on the recognition of their chromatographic patterns. Quantitation is performed using the area of characteristic peaks in the sample and standard using external or internal calibration procedures.

If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be warranted. Suggested cleanup options are given in Section 9.

11.1.6.1PCBs as Aroclors

PCBs are generally reported as Aroclors. The Aroclors have varying levels of PCB congeners with the last two numbers in the Aroclor designation indicating the weight percent of chlorine. For example, AR1221 is 21% chlorine by weight; AR1260 is 60% chlorine by weight. The 12- in the Aroclor designation represents the biphenyl molecule. The exception to this naming convention is AR1016, which is about 42% chlorine by weight. (Note that AR1016 and AR1242 have similar chromatograms - both Aroclors have almost the same weight of chlorine by weight and nearly the same PCB congeners.)

Aroclors are identified by matching the pattern of the sample with standards analyzed under the same analytical conditions. Interference may occur due to the presence of non-target analytes or due to "weathering" of the Aroclor in the environment. The presence of multiple Aroclors will also complicate identification and quantitation. Many matrix interferences may be reduced or eliminated by treating the sample extract with copper, and sulfuric acid, prior to analysis. STL SL SOP EX60 details this procedure.

NOTE: Do not use the acid cleanup on the entire extract if pesticides are also to be reported as many of the pesticides are not stable in acid or strong oxidizer.

When a pattern matching an Aroclor is encountered, it may be quantitated using either the 3-5 characteristic peaks (recommended) or total area response. Total area quantitation should only be used as detailed below. Residues of either AR1016 or AR1260 are quantitated using the average RF/CF determined during initial calibration. The other Aroclors are quantitated against the RF/CF determined from their single-point analysis during initial calibration. Samples should be diluted when the amount of PCB in a sample extract exceeds the calibration range defined in initial calibration. Note that the AR1660 standard defines the working range for all the Aroclors. (i.e. if AR1660 was calibrated from 0.10µg/mL to 5.0µg/ml, and a sample extract was analyzed containing 10µg/ml of AR1232, that extract would require dilution to get the amount of AR1232 to be less than 5.0µg/ml.) If a sample contains any of the single point Aroclors (that is, Aroclors other than AR1016 and AR1260), then that associated standard must be run within 72 hours of the sample to determine retention time shifts and pattern recognition.

In the 3-5 peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of PCB in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 3-5 resulting concentrations are averaged to provide the final result for the sample.

"Weathering" is the loss of part of the Aroclor pattern due to biological or chemical degradation of individual PCBs. When weathering is suspected, try to match the later eluting peaks first. Flag the results for a weathered Aroclor pattern as tentatively identified or make a note in the case narrative if provided.

The presence of multiple Aroclors can be a problem to identify since most Aroclors have at least a few peaks in common. The easiest case would be to have early and late eluting Aroclors present. The most difficult cases will involve the presence of Aroclors with the same relative chlorine level.

NOTE: When choosing individual peaks for quantitation, compare their responses in the sample and standard. If the peaks chosen do not correlate well (i.e. ratios to other peaks are close) between the sample and standard, review the chromatograms for other possible peaks for quantitation.

11.1.5.2 Toxaphene

Toxaphene is a mixture of chlorinated camphenes, which has a complex and characteristic pattern when analyzed by GC-ECD. A single Toxaphene standard is analyzed during the initial calibration for the purpose of pattern identification in samples. When a Toxaphene residue is detected in sample(s), sample analysis is stopped. A calibration curve with at minimum of 5 points bracketing the instrument calibration range for Toxaphene should be analyzed. Alternatively, single points may be prepared with Toxaphene concentrations within 2x the Toxaphene quantity detected in the samples. Generally, the calibration curve option is simpler. After analysis of the Toxaphene standard(s), the samples are re-analyzed using this standard(s) for quantitation. Note that when analysis of Toxaphene-containing samples occurs over an

extended time, the calibration factor should be verified or regenerated every 12 hours for method 8081, and every 24 hours for method 608.

If the sample and standard chromatograms agree well, Toxaphene is quantitated using 5 characteristic peaks (similar to the PCB approach, above).

When Toxaphene is determined using the 5-peak approach, the analyst must take care to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms. It is highly unlikely that the peaks will match exactly, but the analyst should not employ peaks from the sample chromatogram whose relative sizes or areas appear to be disproportionately larger or smaller in the sample compared to the standard.

In the 5-peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of Toxaphene in the standard. A minimum of three peaks must be used to determine the calibration factor. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 5 resulting concentrations are averaged to provide the final result for the sample.

11.1.5.3 Chlordane

Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. The following components are significant: α and γ Chlordane, trans-Nonachlor, Heptachlor, and Heptachlor-epoxide. The α and γ Chlordane isomers are the most prevalent and their detection as single components is a good indicator that Technical Chlordane may be present.

When the GC pattern of the residue resembles that of the Technical Chlordane standard, quantitate Chlordane residues by comparing the area of 3 to 5 major peaks. Heptachlor and heptachlor epoxide should not be included in this quantitation but rather should be quantitated and reported separately.

When a Technical Chlordane residue is detected in sample(s), sample analysis is stopped. A calibration curve with at minimum of 5 points bracketing the instrument calibration range for technical chlordane should be analyzed. Alternatively, single points may be prepared with technical chlordane concentrations within 2x the Technical Chlordane quantity detected in the samples. Generally, the calibration curve option is simpler. After analysis of the technical chlordane standard(s), the samples are re-analyzed using these standard(s) for quantitation. Note that when analysis of technical chlordane-containing samples occurs over an extended time, the calibration factor should be verified or regenerated every 12 hours for method 8081, and every 24 hours for method 608.

NOTE: These procedures are not necessary if the lab is reporting chlordane as the α and γ chlordane isomers, not as the technical product.

11.2 Refer to SOP AN67: *Evaluation of Calibration Curves* for the calculations of sample concentrations.

12.0 QUALITY CONTROL AND DATA ASSESSMENT

12.1 *Analytical Batching*

QC data must be evaluated against the precision and accuracy criteria set forth in the Laboratory Quality Manual and SOP AN02: *Analytical Batching and Evaluation of QC Data*. These criteria are summarized in the SOP Summary included in Appendix C

SOP AN02 also provides guidance for establishing and evaluating QC items to be included in an analytical batch.

12.2 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP CA85: *Nonconformance and Corrective Action Procedures*. CA85 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures.

13.0 METHOD PERFORMANCE

The Reporting Limits (RL), the Method Detection Limits (MDL), and accuracy and precision limits associated with these methods are given in the current revision of the Laboratory Quality Manual prepared by and for STL Savannah.

13.1 Initial and Continuing Demonstration of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP *CA92: Procedure for Initial and Continuing Analyst Demonstration of Capability*.

13.2 Method Detection Limit

The method detection limit must be determined annually for each analyte in accordance with SOP CA90: *Procedures for the Determination of Method Detection Limit (MDL)*.

14.0 PREVENTIVE MAINTENANCE AND TROUBLESHOOTING

Refer to SOP AN53: *Maintenance Procedures for Laboratory Instrumentation* for routine preventive maintenance and the manufacturer's guides for trouble-shooting items.

15.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15.1 Waste Streams Produced by the Method

Excess samples, reagents, and standards must be disposed in accordance with SOP CA70: *Waste Management*.

The following waste streams are produced when this method is carried out:

- Flammable waste (hexane and methanol from extracts, rinsings, and standards) – Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full.
- Excess water samples. Dispose according to characterization on sample disposal sheets. If non-hazardous, dispose down drain/sewer. If hazardous, transfer to hazardous waste department for storage.
- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.
- Methylene chloride extracts. Dispose according to characterization on sample disposal sheets. If non-hazardous, transfer extract to chlorinated waste container. If hazardous, transfer to hazardous waste department for storage.

16.0 REFERENCES

Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846; including Update III. U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

Code of Federal Regulations, Title 40, Part 136; U.S. Government Printing Office: Washington, DC, July 1, 1988.

17.0 TABLES, DIAGRAMS, AND VALIDATION DATA

Appendix A: CHLORINATED PESTICIDES AND PCBs STANDARDS

Routine Targets

Target compound	STD ISMA-1 (ug/mL)	STD ISMA-2 (ug/mL)	STD ISMA-3 (ug/mL)	STD ISMA-4 (ug/mL)	STD ISMA-5 (ug/mL)
TCMX, DCB, OCN (sum)	0.0025	0.0050	0.010	0.020	0.040
g-BHC(Lindane), Heptachlor, Heptachlor epoxide, Endosulfan I, a-BHC, b-BHC, d-BHC, a-Chlordane, g-Chlordane	0.0050	0.010	0.020	0.030	0.050
Dieldrin, p,p'-DDT, Endosulfan II, Endrin aldehyde, Methoxychlor, p,p'-DDE, Endrin p,p'-DDD, Endosulfan sulfate, Endrin ketone	0.010	0.020	0.040	0.060	0.10

DDT/Endrin Breakdown Evaluation Standard

Pesticide Evaluation Standard	CONC (ug/mL)
Endrin,	0.040
P,P'-DDT	0.040

Appendix IX Targets

Target compound	APIX-1 (ug/mL)	APIX -2 (ug/mL)	APIX -3 (ug/mL)	APIX -4 (ug/mL)	APIX -5 (ug/mL)
TCMX, DCB, OCN (sum)	0.0025	0.0050	0.010	0.020	0.040
Isodrin	0.0050	0.010	0.020	0.030	0.050
Chlorobenzilate	0.050	0.10	0.20	0.50	1.0
Kepone	0.025	0.050	0.10	0.20	0.50

APPENDIX B

Technical Chlordane Five-point Curve

STOCK STANDARD	TCHLOR -1	TCHLOR -2	TCHLOR -3	TCHLOR -4	TCHLOR -5
Technical Chlordane	0.10	0.20	0.40	0.60	0.80
TCMX, DCB OCN (surr)	0.0025	0.0050	0.010	0.020	0.040

Toxaphene Five-point Curve

STOCK STANDARD	TOX -1	TOX -2	TOX -3	TOX -4	TOX -5
Toxaphene	0.10	0.20	0.40	1.0	2.0
TCMX, DCB, OCN (surr)	0.0025	0.0050	0.010	0.020	0.040

PCBs as Aroclors

AR1660 Standards

Calibration Std	AR1016 (ug/mL)	AR1260 (ug/mL)	Surrogates (ug/mL)
AR1660-1	0.10	0.10	0.0025
AR1660-2	0.20	0.20	0.0050
AR1660-3	0.50	0.50	0.010
AR1660-4	1.0	1.0	0.020
AR1660-5	2.0	2.0	0.040

Single Point Aroclor Calibration Standards

Calibration Standard	Single Pont Concentration (ug/mL)	Surrogate Concentrations (ug/mL)
AR1221	1.0	0.020
AR1232	1.0	0.020
AR1242	1.0	0.020
AR1248	1.0	0.020
AR1254	1.0	0.020

If required, five point curves for AR1221, AR1232, AR1242, AR1248, and AR1254 are prepared at the same concentrations as the AR1660 curve.

Appendix B - METHOD SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage*	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	None; 4C	1-L amber	7 days	40 days
Soil/ Sediment	None; 4C	500-mL	14 days	40 days
Waste	None; 4C	Glass	14 days	40 days
TCLP	None; 4C	1-L amber	7 days to leach / 7 days (after leaching procedure)	40 days

*Storage temperature is 4°C with control criteria of less than 6°C with no frozen samples

EXTRACTION

Aqueous: Approximately 1L of sample (contents of container) using continuous at pH 5-9 with methylene chloride; exchange to hexane and concentrate to final volume of 10mL

Soil/Solids: Approximately 30g of sample using sonication with 1:1 acetone/hexane or 1:1acetone/methylene chloride; Concentrate to final volume of 10mL in hexane

Wastes: Approximately 1g of sample diluted to final volume of 10mL with hexane

ANALYSIS

Dual capillary columns with dual EC; 2-5uL injection into glass tee or y-splitter; external or internal standard calibration

SURROGATE:	Aqueous (ug/L)	Soils (ug/kg)
Tetrachloro-m-xylene	0.50	15
Decachlorobiphenyl	0.50	15
OCN	0.50	15

608 BATCH QC

Method blank
LCS/LCSD- full target list of single peak analytes @ 0.20ug/L
MS/MSD- full target list of single peak analytes @ 0.20ug/L

8081/8082 BATCH QC

Method blank
LCS - LQM subset
MS/MSD - LQM subset

Parameter	Aqueous (ug/L)	Soils (ug/kg)
Lindane	0.20	6.0
Aldrin	0.20	6.0
Heptachlor	0.20	6.0
Dieldrin	0.50	15
Endrin	0.50	15
p,p'-DDT	0.50	15

Appendix C

The sequence continues until all samples have been analyzed or until the calibration verification fails the acceptance criteria. All sample extract analyses must be bracketed by acceptable verification standards. The default procedure is not to count the QC items in the 20 sample extracts that may be analyzed in the clock; i.e.; the number of sample and QC extracts may exceed 20 but the total number of sample extracts may not exceed 20 and all extracts (samples and QC) must be analyzed within the 12-hour clock for 8081/8082 and a 24-hour clock for 608.

ANALYTICAL SEQUENCE – 608, 8081A, and 8082 (Pesticides and PCBs)

STANDARD / SAMPLES
Initial Calibration - 5 point single peak pesticides 5 point AR1660 (*Note 2) 1 point Toxaphene (*Note 1) 1 point Technical Chlordane (*Note 1) 1 point remaining Aroclors
Instrument Blank
PEVAL - Endrin/p,p'-DDT Breakdown Check (*Note 3)
ICV (Initial Calibration Verification) - 2nd source calibration verification
Up to twenty sample extracts and QC and/or 12 hours for 8000-series Up to twenty sample extracts and QC and/or 24 hours for 600-series
PEVAL - Endrin/p,p'-DDT Breakdown Check (*Note 3)
CCV (Continuing Calibration Verification) - Midpoint single peak pesticides and midpoint AR1660 (*Note 2)
Instrument Blank
Up to twenty sample extracts and QC and/or 12 hours for 8000-series Up to twenty sample extracts and QC and/or 24 hours for 600-series
PEVAL - Endrin/p,p'-DDT Breakdown Check (*Note 3)
CCV (Continuing Calibration Verification) - Midpoint single peak pesticides and midpoint AR1660 (*Note 2)
Instrument Blank

*Note 1 – If Toxaphene or Technical Chlordane is detected in a sample, a full 5-point ICAL must be performed and the sample reanalyzed.

*Note 2 - A mixture of AR1016 and AR1260 will be used to calibrate and verify the response for PCBs. Mid-level standards of the remaining Aroclors must be analyzed every 72 hours for pattern recognition and retention time.

*Note 3 – PEVAL Breakdown Check is not required for PCB-only analyses

Appendix C

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
p,p'-DDT and Endrin breakdown check -required for 8000-series, recommended for 600-series	Initially and every 12 hours	% breakdown of both compounds less than 15%	-re-analyze check solution -perform injector port and/or column maintenance and re-analyze
Initial Calibration- 600-series: 3 point minimum with lowest point at RL 8000-series: 5 point minimum with lowest point at RL	Initially prior to sample analysis, when major instrument maintenance performed, or when CCV fails	**600-series:** 1) RSD of each target <= 10%; OR 2) plot regression curve CC>=0.99 for each target **8000-series:** 1) RSD of each target <= 20%; OR 2) plot regression curve CC>=0.99 for each target	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification (CCV)	After every twenty sample analyses and at the end of the sequence 8000-series: the capping standard must be injected within 12-hours of the PEVAL after ICAL, within 12 hours of the previous CCV 600-series: the capping standard must be injected within 24-hours of the PEVAL after ICAL, within 24-hours of the previous CCV	-Percent difference or drift <= 15% (see SOP for use of "grand mean") -Response of the internal standard must be within a range of +/-50% of the mid-level standard (CCV) in the ICAL	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards

Appendix C

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method blank	Per batch	All targets reported less than RL in LQM	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02
Lab control sample (LCS)	Per batch (If MS/MSD cannot be performed, the LCS must be performed in duplicate)	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02
Matrix spik e(MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02
Internal Standard Response	All samples, method blanks, and QC	-response within a factor of +/–50% of the previous CCV -retention time within window defined by previous CCV	-Evaluate chromatogram and integrations. -Reanalyze or dilute and reanalyze -Flag data
Surrogates	All samples, method blanks, and QC	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02
Dilutions	When extract concentration exceeds calibration range	Report dilution where highest concentration target is in upper half of calibration	-Dilute sample to bring highest concentration into upper half of calibration range. Report all other targets from this dilution. -If lower RL required, prepare dilution 1/10 of dilution that puts highest concentration target into upper half of calibration range.

Appendix C

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Retention time window determination	Annually and with each new column. See guidance in SOP AN66	See guidance in SOP AN66	Use guidance in SOP AN66: *Determination of Retention Time Windows in Gas Chromatographic Analyses*
Demonstration of Capability	Initially and then annually per work group or analyst	Within the 600- or 8000-series method limits (see SOP CA92 and LQM)	-Reanalyze QC sample for the targets that failed to meet the criteria (see SOP CA92)
Method detection limit (MDL)	Annually See SOP CA90	Evaluate data using criteria SOP CA90	-Evaluate data. Check calculations. -Reanalyze MDL samples.

SEVERN TRENT STL

SOP SUMMARY FORM

SOP#: SG45:03.17.04:8	SOP Description: ORGANOCHLORINE PESTICIDES AND PCBs BY GC

Revisions:

___Minor _X_Significant ___Complete Re-write ___New SOP

Summary of Revision(s):

- Revised format to be consistent with current STL Savannah SOP format and NELAC requirements
- Revised safety information to be consistent with current STL Savannah format
- Updated Materials and Apparatus listing
- Added Quality Control, Method Performance and Waste Management information
- Removed Appendix A, retention time table
- Combined 8000-series and 600-series QC information into Appendix B
- Added cap to Grand Mean criteria per SOP AN67
- Added ICV requirement per SOP AN67
- Added requirement that alternate surrogate to DCB must be evaluated if AR1268 is detected
- Removed total area quantitation option for PCBs and Toxaphene, no longer performed
- Removed option NOT to perform capping CCV for internal standard analyses. Although method 8000 allows this option, Method 8081/8082 explicitly states that capping CCV must be performed.
- Removed reference to Separatory Funnel Extractions, no longer performed
- Removed reference to Permanganate clean-ups, no longer performed

IDOCs Required:	___Yes	_X_ No	__NA
MDLs Required:	___Yes	_X_ No	__NA

SOP Implementation Date: 04.17.04

Target Training Completion Date: 04.17.04

Approved by: Andrea Sal

Title: Quality Assurance Manager

Date: 03/22/04

Division Approval:

Title: Laboratory Director

Date: 3/22/04

SOLUTIA - 202

Kenneth
Bardo/R5/USEPA/US
07/13/2005 03:49 PM

To sdsmit@solutia.com
cc bsyare@solutia.com
bcc GARY CYGAN/R5/USEPA/US@EPA

Subject Plume Stability Plan

Steve - This e-mail is a follow-up to our discussion yesterday regarding the proposed screen depths for the 17 monitoring wells described in the *Plume Stability Monitoring Plan* submitted on July 5, 2005. All monitoring wells are proposed to have 5-foot screens at the top of rock (TOR). The basis for proposing this screen interval was to monitor the largest plume in areal extent which occurs in the deep hydrogeological unit (DHU).

Solutia initially proposed to screen monitoring wells at various intervals based on the highest contaminant concentration found at depth. Generally, highest concentrations are found in the shallow hydrogeological unit (SHU) and middle hydrogeological unit (MHU) near source areas. With distance from the source, highest concentration are more typically found in the DHU but not necessarily at the TOR. At the river, it is EPA's understanding that the the main release to the river is from the MHU (monitoring the MHU at the river would best reflect current discharges). So depending on location, highest concentrations in the aquifer vary with depth and Solutia's initial approach to screen the most contaminated zone (and thus having variable screen depths) made sense. Monitoring the most contaminated zone would also most readliy reflect improvements in groundwater quality as source areas are remediated.

The proposed screen depths are not consistent with this approach or the model using EVS, Version 7.9.2. For example, Figure No. 2.8 shows that the modelled highest concentrations of MCB at wells 5 and 8 are significantly above the TOR but 5-foot screens are proposed at the TOR. Also, real data from groundwater samples taken at various depths from transect borings AA-GWM-S1, AA-GWM-S2, and AA-GWM-S3 shows that highest MCB concentrations are 70', 25', and 20' respectively above the TOR.

As we discussed yesterday, Solutia should evaluate real data from wells or borings in the immediate vicinity of the 17 proposed monitoring wells. Much historical data exists (e.g., see CA750 report) and many of the site-wide wells are currently being sampled as part of the CERCLA investigation. We believe that real, not modelled data would be more appropriate in determining screen depths. To ensure adequate coverage of the most contaminated zone, screen lengths of up to 10' may be acceptable.

An evaluation of existing data should be conducted to provide and justify screen depths at the 17 proposed well locations. Please provide this information by the first week of August. A meeting the week of August 8 can be held to discuss your results and finalize any outstanding issues regarding the *Plume Stability Monitoring Plan*.

I will be returning to the office on July 25th if you have any questions. - Ken

SOLUTIA - 204

www.ep

Rod R. Blag

Return to Search Criteria

Groundwater Ordinances Reviewed for Use As Environmental Institutional Controls Sauget-Municipality

County	Review Completion Date	Division of Legal Counsel Ref #	Citation	Ordinance	Comments	Ordinance Document
St. Clair	8/3/2005	R05070602		99-5, as amended by 05-03	Ordinance approved.	R05070602.pdf

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Run Date :7/6/2005

DLC Assignment Form

Assignment ID :4329
Subject :Sauget/Big River Zinc
Subject Type :Ordinance Review
DLC In Date :7/6/2005
DLC File No. :
Correspondence No. :R05070602

DLC Completed Date. :

Assigned Staff:
Flowers, Stephanie Attorney
McCaslin Bureau Requestor

Project Details:
Status Issued Date: 7/6/2005 Due Date: 8/5/2005
Please review groundwater ordinance for Village of Sauget

Comments:

STATE OF ILLINOIS)
COUNTY OF ST. CLAIR) ss
VILLAGE OF SAUGET

CERTIFICATE

I, the undersigned, Janice D. Delaney, do hereby CERTIFY that:

1. I am the duly qualified and acting Village Clerk of the Village of Sauget, an Illinois municipal corporation and home rule unit situated in St. Clair County, Illinois. As such, I have the care and custody of its minutes, ordinances, resolutions, and other documents and its official seal.

2. On the 12th day of October, 1999, a meeting of the President and Board of Trustees of said VILLAGE was duly and properly convened, held and conducted. If required, due proper and timely notice was given.

3. At said meeting, a quorum was present throughout.

4. At said meeting, that certain document entitled:
" AN ORDINANCE PROHIBITING THE USE OF GROUNDWATER AS A POTABLE WATER SUPPLY BY THE INSTALLATION OR USE OF POTABLE WATER SUPPLY WELLS OR BY ANY OTHER METHOD. "

Ordinance No. 99-5 Resolution No. _____

was duly and properly adopted by the President and Board of Trustees of said VILLAGE and was thereupon duly approved by the President of said VILLAGE.

5. A true, correct and complete copy of said document is attached hereto and, by this reference, incorporated herein and made a part hereof.

6. The original of said document is in my possession and control as said Village Clerk.

7. The provisions of said document have not been altered, changed or amended in any manner whatever and are now in full force and effect.

Whereof, I have hereunto set my hand and affixed the official seal of said VILLAGE on this 14th day of June, 20 05.

(SEAL)

Janice D. Delaney
Village Clerk
Village of Sauget, Illinois

ORDINANCE NO. 99-5

AN ORDINANCE PROHIBITING THE USE OF GROUNDWATER AS A POTABLE WATER SUPPLY BY THE INSTALLATION OR USE OF POTABLE WATER SUPPLY WELLS OR BY ANY OTHER METHOD

WHEREAS, certain properties in the Village of Sauget, Illinois, have been used over a period of time for commercial/industrial purposes; and

WHEREAS, because of said use, concentrations of certain chemical constituents in the groundwater beneath the Village may exceed Class I groundwater quality standards for potable resource groundwater, as set forth in 35 Illinois Administrative Code Part 620, or Tier I residential remediation objectives, as set forth in 35 Ill. Adm. Code Part 742; and

WHEREAS, the Village of Sauget desires to limit potential threats to human health from groundwater contamination while facilitating the redevelopment and productive use of properties that are the source of said chemical constituents;

NOW, THEREFORE, BE IT ORDAINED BY THE VILLAGE COUNCIL IN THE VILLAGE OF SAUGET, ILLINOIS:

Section One: Use of groundwater as a potable water supply prohibited.

The use or attempted use of groundwater from within the corporate limits of the Village as a potable water supply by the installation or drilling of wells or by any other method is hereby prohibited.

Section Two: Penalties

Any person violating the provisions of this ordinance shall be subject to a fine of up to ~~$500~~ for each violation.

Section Three: Definitions.

"Person" is any individual, partnership, co-partnership, firm, company, limited liability company, corporation, association, joint stock company, trust, estate, political subdivision, or any other legal entity, or their representatives, agents or assigns.

"Potable water" is any water used for human or domestic consumption, including, but not limited to, water used for drinking, bathing, swimming, washing dishes, or preparing foods.

Section Four: Repealer.

All ordinances or parts of ordinances in conflict with this ordinance are hereby repealed insofar as they are in conflict with this ordinance.

Section Five: Severablity.

If any provision of this ordinance or its application to any person or under any circumstances is adjudged invalid, such adjudication shall not affect the validity of the ordinance as a whole or of any portion not adjudged invalid.

Section Six: Effective Date.

This ordinance shall be in full force and effect from and after its passage, approval and publication, as required by law.

INTRODUCED AND READ FOR THE FIRST TIME: October 12, 1999

READ FOR THE SECOND TIME:
(under suspension of rules): October 12, 1999

READ FOR THE THIRD TIME:
(under suspension of rules): October 12, 1999

ADOPTED AND ENACTED: October 12, 1999

ROLL CALL VOTE:
Ayes: Adele, McDaniel, Rich, Cates, Thornton, Sauget
Nays: NONE
Absent: NONE
Unfilled Vacancy:

APPROVED: October 12, 1999

APPROVED:

Paul Sauget
President (mayor) Pro Temore

ATTEST:

Betty Long Wilson
Village Clerk

STATE OF ILLINOIS)
COUNTY OF ST. CLAIR) ss
VILLAGE OF SAUGET

CERTIFICATE

I, the undersigned, Janice D. Delaney, do hereby CERTIFY that:

1. I am the duly qualified and acting Village Clerk of the Village of Sauget, an Illinois municipal corporation and home rule unit situated in St. Clair County, Illinois. As such, I have the care and custody of its minutes, ordinances, resolutions, and other documents and its official seal.

2. On the 10th day of May, 2005, a meeting of the President and Board of Trustees of said VILLAGE was duly and properly convened, held and conducted. If required, due proper and timely notice was given.

3. At said meeting, a quorum was present throughout.

4. At said meeting, that certain document entitled:
" AMENDING ORDINANCE NO. 99-5 "AN ORDINANCE PROHIBITING THE USE OF GROUNDWATER AS A POTABLE WATER SUPPLY BY THE INSTALLATION OR USE OF POTABLE WATER SUPPLY WELLS OR BY ANY OTHER METHODS". "

Ordinance No. 05-03 Resolution No. _____

was duly and properly adopted by the President and Board of Trustees of said VILLAGE and was thereupon duly approved by the President of said VILLAGE.

5. A true, correct and complete copy of said document is attached hereto and, by this reference, incorporated herein and made a part hereof.

6. The original of said document is in my possession and control as said Village Clerk.

7. The provisions of said document have not been altered, changed or amended in any manner whatever and are now in full force and effect.

Whereof, I have hereunto set my hand and affixed the official seal of said VILLAGE on this 6th day of June, 20 05.

(SEAL)

Janice D. Delaney
Village Clerk
Village of Sauget, Illinois

VILLAGE OF SAUGET
ORDINANCE NO. _05-03
AMENDING ORDINANCE NO. 99-5 "AN ORDINANCE PROHIBITING THE USE OF GROUNDWATER AS A POTABLE WATER SUPPLY BY THE INSTALLATION OR USE OF POTABLE WATER SUPPLY WELLS OR BY ANY OTHER METHODS

WHEREAS, the Village of Sauget Board of Trustees passed ordinance No. 99-5 which prohibited the use of groundwater as a potable water supply but failed to clearly indicate that said prohibition extends to the Village itself, as a municipal body; and,

WHEREAS, it is necessary to make this clarification to meet certain residential remedial requirements of the Illinois Environmental Protection Agency.

NOW THEREFORE BE IT HEREBY ORDAINED BY THE BOARD OF TRUSTEES OF THE VILLAGE OF SAUGET, ST. CLAIR COUNTY, ILLINOIS AS FOLLOWS:

1. Section 1 Ordinance No. 99-5 is amended by the addition of a final sentence to Section 1 as follows:" This prohibition expressly includes the Village of Sauget as a municipal body."
2. Section 2 All ordinances or parts of ordinances is conflict with this ordinance are hereby repealed insofar as they are in conflict with this ordinance.
3. Section 3 If any provision of this ordinance or its application to any person or under any circumstances is adjudged invalid, such adjudication shall not affect the validity of the ordinance as a whole or of any portion not adjudged invalid.
4. Section 4 This ordinance shall be in full force and effect from and after its passage, approval and publication, as required by law.

INTRODUCED AND READ FOR THE FIRST TIME: May 10, 2005

READ FOR THE SECOND TIME: May 10, 2005
(under suspension of rules)

READ FOR THE THIRD TIME: May 10, 2005
(under suspension of rules)

ADOPTED AND ENACTED: May 10, 2005

ROLL CALL VOTE:

	AYE	NAY	ABSENT
ADELE	X		
MCDANIEL	X		
WORS			X
CATES			X
THORNTON	X		
NICHOLSON	X		

APPROVED: May 10, 2005

APPROVED: VILLAGE OF SAUGET

BY: _____
Richard Sauget, Jr. President

ATTEST:

_____
Village Clerk
VILLAGE OF SAUGET, ILLINOIS

SOLUTIA - 205

Kenneth Bardo/R5/USEPA/US
08/16/2005 05:42 PM

To "Yare, Bruce S" <bsyare@solutia.com>
cc GARY CYGAN/R5/USEPA/US@EPA, "Williams, Richard S" <rswill1@solutia.com>, "Smith, Steven D" <sdsmit@solutia.com>
bcc George Hamper/R5/USEPA/US@EPA
Subject Re: WGK - Plume Stability Monitoring Well Screen Depths

Bruce - This e-mail responds to the proposed well screen depths presented below in your August 2 e-mail and provides final comments on the Plume Stability Monitoring Plan submitted on July 5, 2005. The comments provided here were also discussed at our August 10th meeting in Chicago. The meeting was very productive and I hope we were able to resolve all outstanding issues. Upon further discussion within EPA regarding the use of fully penetrating screens for sampling, we are not confident that sampling would be representative. This is further discussed in Comment #5 below. The other comments were discussed at the meeting and are memorialized here. We look forward to finalizing the plan and getting the program underway as required by the CA750 Environmental Indicator determination.

1) We agree that the monitoring wells will be constructed of stainless steel with 5-foot screens. Low-flow sampling will be used to obtain groundwater samples.

2) We generally agree with the screen depths proposed in your 8/2/05 e-mail, except as discussed below. However, the use of bgs elevations may cause errors to occur in screen placement since the nearest well used to determine the bgs elevation may vary somewhat in elevation. We calculated the AMSL target elevation level for each new well based on the surface elevations of the nearest well provided in Table A-1 of Appendix A of the plan. Solutia should confirm these values (values in red need further evaluation and are discussed in comments below). The well screen should be placed 2.5-feet above and below the AMSL target elevation.

MW-1 = 370' AMSL MW-2 = 338' AMSL MW-3 = 339' AMSL MW-4 = 307' AMSL MW-5 = 382' AMSL MW-6 = 296' AMSL
MW-7 = 297' AMSL MW-8 = 340' AMSL MW-9 = 301' AMSL MW-10 = 304' AMSL MW-11 = 304' AMSL MW-12 = 310.5' AMSL
MW-13 = 297' AMSL MW-14 = 298.5' AMSL MW-15 = 298.5' AMSL MW-16 = 302' AMSL MW-17 = 293' AMSL

3) For MW-5, a 20-foot thickness (18-38 ft. bgs) was determined. Nearby wells show that highest concentrations were detected at 15-18' at DNAPL-K8 and that nearby wells GM-32, GM-33, and GM-34 are highly contaminated and are apparently screened from 9-24' bgs. The well log for GM-32 shows the screen elevation to be from approx. 385 to 400' AMSL. These four nearby data points appear to show a more contaminated zone above that determined by using data from GM-17A, B, C. Confirm the data discussed here, document, and adjust the AMSL target elevation of 382' upward as necessary.

4) We discussed the need for at least two discrete monitoring levels at MW-14, MW-15, and MW-16. While highest contaminant concentrations may be deep at approximately 300' AMSL, monitoring only this zone would not be representative of groundwater quality and mass loading discharging to the Mississippi River. For example, the base of the river appears to intersect the upper portion of the MHU and the most contaminated sediment was found to occur only approximately 150' from the riverbank. This would imply that discharge from some portion of the MHU would be important to monitor and would be most representative of groundwater quality directly discharging to the river north of Site R. Solutia discussed the use of fully penetrating screens from 280' to 360' AMSL in the well and low flow sampling to measure this mass loading from the MHU. EPA has evaluated this proposal but is concerned that a fully penetrating screen and low flow sampling would not provide true representation of the discrete discharge from the MHU that is required to be monitored for plume stability. In all cases at RCRA facilities, Region 5 requires the use of nested wells to monitor discrete zones. In addition, proper characterization of the discrete zone of the MHU that should be monitored can be performed prior to completion of the screened elevation. Therefore at MW-14 and MW-15, EPA requests that two additional wells at each location be

installed that are screened within the MHU at a screen elevation to be determined. Based on available data from groundwater sampling near the riverbank and river hydrographic surveys, EPA estimates appropriate screen elevations to be in the range of 360' to 370' AMSL.

5) Since additional monitoring is being performed under the CERCLA program at Site R, this additional data can be used to supplement proposed deep monitoring to be performed at MW-17. Solutia should ensure that Site R groundwater, surface water, and sediment monitoring is considered, where applicable, in the evaluation of plume stability.

6) Certain figures provide in the plume stability monitoring plan are inaccurate. Specifically, Figures 2-1, 2-2, 2-3, 2-5, and 2-7 do not accurately portray the data presented in Table A-1 of Appendix A for TRA6-GPC. Significant concentrations of benzene, chlorobenzene, chloroanilines, dichlorobenzenes, and BTEX were found but all figures indicate concentrations of these compounds were <1 ug/l. These figures should be revised to accurately portray the concentrations detected at TRA6-GPC. Alternatively, the figures may be eliminated and be replaced by the data in your 8/2/05 e-mail in support of the proposed well screens depicted in Figure 2.8.

7) Section 2.3 of the plan indicates that a separate workplan will be submitted to address PCBs in groundwater at the Former PCB Manufacturing Area. We agree that the workplan should be submitted as part of the interim measure investigations ongoing at the site related to source controls. Solutia should propose a timeframe for submitting the workplan based on current and projected IM activities at the PCB Manufacturing Area. Please submit a proposed date for the workplan to EPA and the basis for that date by August 31.

8) Tables 4.1 and 4.2 show that PCB analysis will be for Aroclors using Method 8082 but Section 3.3 proposes analysis for homologs using Method 680. Appendix D list the PCB analysis methods as 608/8081/8082. Ensure that all discussion of PCB analyses in the plan are consistent and that PCB homologs will be determined using Method 680.

9) At this time, EPA believes that the schedule presented in Section 5.0 can be met. Wells should be installed as soon as possible to initiate sampling in October. Please update EPA with progress in meeting the schedule or any need to modify the schedule.

A revised Plume Stability Monitoring Plan that addresses these comments should be submitted to EPA within 30 days. If we need to discuss any of these comments further, please let me know. - Ken

"Yare, Bruce S" <bsyare@solutia.com>

"Yare, Bruce S" <bsyare@solutia.com>
08/02/2005 02:51 PM

To

Subject WGK - Plume Stability Monitoring Well Screen Depths

Ken and Gary

Proposed screen depths for the W.G. Krummrich Plume Stability Monitoring Plan are given in separate tables in the attached Excel file (KR073105). These screen depths, which are based on the highest detected groundwater concentrations in the nearest vertical profile to the plume stability monitoring well location, are summarized below:

- MW-1 43 ft bgs
- MW-2 73 ft bgs
- MW-3 72 ft bgs

- MW-4 92.5 - 102.5 ft bgs
- MW-5 18 - 38 ft bgs
- MW-6 110 ft bgs
- MW-7 119 ft bgs
- MW-8 73 ft bgs
- MW-9 104 ft bgs
- MW-10 104 ft bgs
- MW-11 104 ft bgs
- MW-12 100 ft bgs
- MW-13 114 ft bgs
- MW-14 112 ft bgs
- MW-15 112 ft bgs
- MW-16 120 ft bgs
- MW-17 127 ft bgs

Plume stability monitoring wells PSMW-1, 2, 3, 4 and 5 are located in source areas and, as a consequence, selected screen depths are in the lower portion of the SHU and the MHU. The MHU will also be screened at PSMW-8 because VOC and SVOC concentrations are highest in this hydrogeologic unit, probably as a result of past spills and leaks from the Former Chlorobenzene Storage Area. Screens will be installed in the DHU at PSMW-6, 7, 9, 10, 11, 12, 13, 14, 15, 16 and 17. These monitoring wells are located downgradient of the Plant Process Area where the highest groundwater concentrations are found in the deeper portions of the alluvial aquifer.

Screen depths generally get deeper with increasing distance from the W.G. Krummrich facility downgradient boundary. Screen depths at PSMW-6, 7, 8 and 9, located at the downgradient boundary of the plant, average 101.5 feet and range from 73 to 119 ft bgs. Monitoring wells PSMW-10, 11, 12 and 13, which are located approximately half way between the plant boundary and the Mississippi River, have an average screen depth of 104.5 ft bgs and range from 100 to 114 ft bgs. At the Mississippi River, PSMW-14, 15, 16 and 17 have an average depth of 117.75 ft bgs and range from 112 to 127 ft bgs.

This screen depth pattern reflects high concentrations in shallow groundwater in the source areas and high concentrations in deep groundwater in the discharge area as groundwater migrates from the plant and discharges to the Mississippi River.

I hope this screen depth selection approach meets your expectations better than the krieging approach.

Please accept my apologies for not finishing this last week. Time got away from me.

See you next Wednesday, August 10, 2005, to discuss.

Bruce

(Excel File deleted - KB)

SOLUTIA - 206

Kenneth Bardo/R5/USEPA/US
09/13/2005 01:18 PM

To "Smith, Steven D" <sdsmit@solutia.com>
cc George Hamper/R5/USEPA/US@EPA, briller_dan@bah.com
bcc Nabil Fayoumi/R5/USEPA/US@EPA
Subject Re: FW: WGK - PCB Thermal Treatability Test

Steve Smith - I am discouraged to hear at this late date that appropriate samples have not yet been obtained for conducting the treatability tests. It has been a month since we were informed at an August 10th meeting that soil samples from the MCB/DCB source areas were not comparable to the historical high concentrations. At that time, Solutia represented that the PCB soil sample concentrations were OK and were actually higher than historical high concentrations (i.e., 22,000 ppm). On or about August 12, we responded that spiking the soil samples with MCB/DCB was appropriate and should be performed. At that time, it was our understanding that the spiking would occur and that the treatability tests would move forward.

Your e-mail of September 9th now states that the PCB soil samples are also well below the historical high concentration. Any additional sampling to obtain results comparable to historical concentrations will further delay the start of the In Situ Thermal Desorption (ISTD) treatability test. We have been discussing the implementation of the treatability tests at the PCB and MCB/DCB source areas since early-2005 (approx. 9 months). I am disappointed by the progress that has been made since then to advance the treatability tests. The expected time frame for completing the tests and submittal of reports is September 23, 2005, for the ISTD treatability test (PCBs) and December 23, 2005, for the Enhanced Aerobic Bioremediation treatability test (MCB/ DCB). Based on your e-mail, it is not readily apparent whether MCB/DCB spiking has occurred (even though we concurred with the procedure one month ago) and it is obvious that the ISTD treatability test will not be completed on time.

As you know, our November 18, 2004, letter highlighted major deficiencies in the Solutia CMS . Rather than EPA proceeding with the available information and initiating a proposal for final corrective measures, we agreed to proceed with interim measures in the form of treatability studies and potential source controls to rapidly address and stabilize ongoing releases at the Solutia facility. However the excessive time it has taken to begin the treatability tests to evaluate the feasibility of engineered source controls is inconsistent with our approach for interim measures/stabilization. We are concerned that Solutia's effort has not been commensurate with EPA's expectation and priority for proceeding with IMs and stabilizing source areas.

Action to Complete Treatability Tests: Data submitted by Solutia, shows that the maximum concentration of PCBs found at 16 sample locations at the Former PCB Manufacturing Area range from 10.6 to 22,100 mg/kg. The median is 1,750 ppm and the mean is 3,196 mg/kg. At 2,447 mg/kg, the PCB concentrations of the original sample obtained for the treatability test is fairly representative of the typical PCB concentrations found in the area. However, as provided for in the workplan, the ISTD test was proposed to be performed on the maximum concentrations detected in the area. Therefore, Solutia should immediately re-mobilize in the field and obtain additional soil samples from former sample locations S0825 and S0835. Individual homgenization and separation into bench-test aliquots should be performed on soil from each sample location. The treatability tests should then be perfomed on the homogenized sample with the highest concentration of PCBs. If neither sample location provides a higher concentration than 2,447 mg/kg, the original soil sample should be used in the treatability tests.

If you have any questions, please let me know. - Ken Bardo

"Smith, Steven D" <sdsmit@solutia.com>

"Smith, Steven D" <sdsmit@solutia.com>
09/09/2005 02:40 PM

To

Subject FW: WGK - PCB Thermal Treatability Test

Ken - May we proceed with the ISTD test, or would you rather we go back to the field and try to find a sample with higher PCB concentrations?

Steve

From: Yare, Bruce S

Sent: Thursday, September 08, 2005 5:57 PM

To: Smith, Steven D

Cc: Williams, Richard S

Subject: WGK - PCB Thermal Treatability Test

Steve

We received results from the field baseline analyses of the ISTD Treatability Study samples yesterday. Results are summarized below:

- **Sample SO825 (Unsaturated SHU)**
  - **PCB**
    - **Actual 2,447 mg/kg**
    - **Target 22,100 mg/kg**
- **Sample SCTB67 & DNAPL-K-4@9' (Unsaturated SHU)**
  - **MCB**
    - **Actual 870 mg/kg**
    - **Target 23,000 mg/kg**
  - **DCB**
    - **Actual 117 mg/kg**
    - **Target 13,850 mg/kg**
- **Sample DNAPL-K-4@16.5' (Saturated SHU)**
  - **MCB**
    - **Actual 210 mg/kg**
    - **Target 1,600 mg/kg**
  - **DCB**
    - **Actual 1,270 mg/kg**
    - **Target 2,950 mg/kg**

None of the samples achieved target (historical high) concentrations even though they were collected at the same depths and locations as the historical high concentrations.

MCB and DCB thermal treatability test samples can be spiked so that they equal or exceed historical high concentrations.

The PCB thermal treatability test samples can not be spiked to achieve historical high concentrations

because PCBs are no longer available in quantities large enough to spike the volume of soil needed for the treatability tests.

We have two choices on how to proceed:

- Perform the PCB thermal treatability test on the 2,447 mg/kg sample
- Collect additional samples until we match or exceed the historical high concentration.

I am reluctant to proceed with the treatability tests because the 2,447 mg/kg sample concentration is a factor of 9 lower than the historical high. The Work Plan says that the tests will be performed on samples with the historical high concentration.

Returning to the field to try an find a sample with a concentration equal to or greater than the historical high concentration will extend the duration of the ISTD Treatability Study. Finding the appropriate sample location will be a trial and error process of unknown duration. Once we find a sampling location that matches the historical high concentration, we need to re-mobilize Prosonics in order to get the large volume of sample needed for the treatability tests.

We are proceeding with the MCB/DCB thermal treatability tests. I believe we need some guidance from the Agency on how to proceed with the PCB thermal treatability tests.

Bruce

This electronic mail message is intended exclusively for the individual or entity to which it is addressed. This message, together with any attachment, may contain Solutia confidential and privileged information. The recipient is hereby put on notice to treat the information as confidential and privileged and to not disclose or use the information except as authorized by Solutia. Any unauthorized review, printing, retention, copying, disclosure, distribution, retransmission, dissemination or other use of, or taking of any action in reliance upon, this information by persons or entities other than the intended recipient is prohibited. If you received this message in error, please immediately contact the sender by reply email and delete all copies of the material from any computer. Thank you for your cooperation.

SOLUTIA - 207

DEPARTMENT OF THE ARMY
ST. LOUIS DISTRICT, CORPS OF ENGINEERS
1222 SPRUCE STREET
ST. LOUIS, MISSOURI 63103-2833

REPLY TO
ATTENTION OF:

September 16, 2005

Operations, Readiness &
Regulatory Division
File Number: MVS-2005-44946

Mr. George Hamper, P.E.
U.S. Environmental Protection Agency
Region 5
77 West Jackson Boulevard
Chicago, IL 60604-3590

Dear Mr. Hamper:

This letter is in reference to correspondence received July 25, 2005, requesting permission to collect soil and sediment samples along the Illinois bank of the Mississippi River. We have reviewed your request to conduct boring activities to determine the presence of contaminants in sediment downstream of historical discharges from industrial facilities located in Sauget, Illinois. Specifically, the boring is needed to determine further action concerning non-permitted discharges in the area. Core samples will be collected from Geoprobe and Vibrocore sampling tools approximately 2 inches in diameter. The collection occurs from Mississippi River mile 190 to 166. The project occurs in St. Clair County, Illinois.

Based upon a review of the U.S. Geological Survey 7.5-minute topographical map, National Wetland Inventory Maps, and information provided by the applicant, we determined that the Mississippi River does possess an ordinary high water mark at this location and would be considered a jurisdictional water of the United States. Therefore, the placement of fill material below the ordinary high water elevation requires a permit from this office.

Section 404 of the Clean Water Act assigns responsibility to the Secretary of the Army to administer a permit program to regulate the excavation or placement of dredged or fill material in waters of the United States. The excavation or placement of any dredged or fill material in waters of the United States below ordinary high water elevation or in wetlands, must be authorized by a Section 404 permit. Your project involves discharges of fill material into waters of the United States, therefore Section 404 authorization is required.

Section 10 of the Rivers and Harbors Act assigns responsibility to the Secretary of the Army to regulate virtually any construction, excavation, or deposition of materials in, over, or under navigable waters of the United States, or any work that could affect the course, location, condition, or capacity of those waters. This ensures that no activity shall impede or interrupt commercial navigation.

The Corps of Engineers has determined that this activity will have no effect on endangered species, and is authorized under Section 404 of the Clean Water Act by an existing Department of the Army nationwide permit as described in the January 15, 2002, Federal Register, Issuance of Nationwide Permits; Notice (67 FR 2078), Appendix A (B)(3). **This permit verification is valid until the nationwide permit (NWP) is modified, reissued, or revoked. All of the existing NWPs are scheduled to be modified, reissued, or revoked prior to March 18, 2007. It is incumbent upon you to remain informed of changes to the NWPs. We will issue a public notice when the NWPs are reissued. Furthermore, if you commence or are under contract to commence this activity before the date that the relevant nationwide permit is modified or revoked, you will have twelve (12) months from the date of the modification or revocation of the NWP to complete the activity under the present terms and**

conditions of this nationwide permit. Enclosed is a copy of the nationwide permit and conditions and management practices with which you must comply.

In accordance with General Condition number 14 of the Nationwide Permits, a compliance certification (Attachment A of this package) must be completed within 30 days of project completion or the permit issuance will be revoked and considered null and void.

The Illinois Environmental Protection Agency (IEPA) has issued Section 401 water quality certification for all projects, which qualify for this nationwide permit. Therefore, individual certification is not required.

The jurisdictional determination for this project is considered an **approved Jurisdictional Determination** in accordance with final regulations published on March 28, 2000(65 FR 16485-16503). Enclosed is a ***Notification of Administrative Appeal Options and Process and Request for Appeal*** for your consideration and use. This form allows you to request an appeal of a jurisdictional determination, however, a preliminary determination cannot be appealed. The jurisdictional determination is valid for a period of five years from the date of this letter unless new information warrants revision of this determination before the expiration date.

This determination is applicable only to the permit program administered by the Corps of Engineers. It does not eliminate the need to obtain other Federal, state or local approvals before beginning work.

You are reminded that the permit is based on submitted plans. Variations from the submitted plans shall constitute a violation of Federal law and may result in the revocation of the permit. This permit authorization is valid for a period of two years for the date of this letter.

If your project does not satisfy all conditions and management practices as indicated, please contact me at (314) 331-8811. Please include the identification number MVS-2005-44946 with any inquiries about this project.

Sincerely,

Alan Edmondson
Rivers Permits Section
Regulatory Branch

Enclosures

Copy Furnished: (w/o enclosures)

Dan Briller, Booz Allen Hamilton, Suite 1700, 225 West Wacker Dr., Chicago, IL 60606-1228
Mr. Robert Dalton, Illinois Department of Natural Resources
Mr. James Allison, Illinois Environmental Protection Agency

ATTACHMENT A

COMPLETED WORK CERTIFICATION

Date of Issuance: September 16, 2005

File Number: MVS-2005-44946

Name of Permittee: Mr. George Hamper, P.E., U.S. Environmental Protection Agency

River Basin/County/State: Mississippi River/St. Clair/Illinois

Upon completion of this activity authorized by this permit and any mitigation required by the permit, sign this certification and return it to the following address:

U.S. Army Corps of Engineers
Attn: Regulatory Branch (CO-F)
1222 Spruce Street
St. Louis, Missouri 63103-2833

(Please note that your permitted activity is subject to a compliance inspection by a U.S. Army Corps of Engineers representative. If you fail to comply with this permit, you are subject to permit suspension, modification or revocation.)

I hereby certify that the work authorized by the above referenced permit has been completed in accordance with the terms and conditions of the said permit, and required mitigation was completed in accordance with the permit conditions.

Kenneth S. Barko — November 1, 2005

Signature of Permittee **Date**

for George Hamper

NOTIFICATION OF ADMINISTRATIVE APPEAL OPTIONS AND PROCESS AND REQUEST FOR APPEAL

Applicant: U.S. Environmental Protection Agency, Attn: Mr. George Hamper, P.E	File Number: MVS-2005-44946	Date: 09/16/2005

Attached is:		See Section below
	INITIAL PROFFERED PERMIT (Standard Permit or Letter of permission)	A
	PROFFERED PERMIT (Standard Permit or Letter of permission)	B
	PERMIT DENIAL	C
X	APPROVED JURISDICTIONAL DETERMINATION	D
	PRELIMINARY JURISDICTIONAL DETERMINATION	E

SECTION I - The following identifies your rights and options regarding an administrative appeal of the above decision. Additional information may be found at http://usace.army.mil/inet/functions/cw/cecwo/reg or Corps regulations at 33 CFR Part 331.

A: INITIAL PROFFERED PERMIT: You may accept or object to the permit.

- ACCEPT: If you received a Standard Permit, you may sign the permit document and return it to the district engineer for final authorization. If you received a Letter of Permission (LOP), you may accept the LOP and your work is authorized. Your signature on the Standard Permit or acceptance of the LOP means that you accept the permit in its entirety, and waive all rights to appeal the permit, including its terms and conditions, and approved jurisdictional determinations associated with the permit.
- OBJECT: If you object to the permit (Standard or LOP) because of certain terms and conditions therein, you may request that the permit be modified accordingly. You must complete Section II of this form and return the form to the district engineer. Your objections must be received by the district engineer within 60 days of the date of this notice, or you will forfeit your right to appeal the permit in the future. Upon receipt of your letter, the district engineer will evaluate your objections and may: (a) modify the permit to address all of your concerns, (b) modify the permit to address some of your objections, or (c) not modify the permit having determined that the permit should be issued as previously written. After evaluating your objections, the district engineer will send you a proffered permit for your reconsideration, as indicated in Section B below.

B: PROFFERED PERMIT: You may accept or appeal the permit

- ACCEPT: If you received a Standard Permit, you may sign the permit document and return it to the district engineer for final authorization. If you received a Letter of Permission (LOP), you may accept the LOP and your work is authorized. Your signature on the Standard Permit or acceptance of the LOP means that you accept the permit in its entirety, and waive all rights to appeal the permit, including its terms and conditions, and approved jurisdictional determinations associated with the permit.
- APPEAL: If you choose to decline the proffered permit (Standard or LOP) because of certain terms and conditions therein, you may appeal the declined permit under the Corps of Engineers Administrative Appeal Process by completing Section II of this form and sending the form to the division engineer. This form must be received by the division engineer within 60 days of the date of this notice.

C: PERMIT DENIAL: You may appeal the denial of a permit under the Corps of Engineers Administrative Appeal Process by completing Section II of this form and sending the form to the division engineer. This form must be received by the division engineer within 60 days of the date of this notice.

D: APPROVED JURISDICTIONAL DETERMINATION: You may accept or appeal the approved JD or provide new information.

- ACCEPT: You do not need to notify the Corps to accept an approved JD. Failure to notify the Corps within 60 days of the date of this notice, means that you accept the approved JD in its entirety, and waive all rights to appeal the approved JD.
- APPEAL: If you disagree with the approved JD, you may appeal the approved JD under the Corps of Engineers Administrative Appeal Process by completing Section II of this form and sending the form to the division engineer. This form must be received by the division engineer within 60 days of the date of this notice.

E: PRELIMINARY JURISDICTIONAL DETERMINATION: You do not need to respond to the Corps regarding the preliminary JD. The Preliminary JD is not appealable. If you wish, you may request an approved JD (which may be appealed), by contacting the Corps district for further instruction. Also you may provide new information for further consideration by the Corps to reevaluate the JD.

SECTION II - REQUEST FOR APPEAL or OBJECTIONS TO AN INITIAL PROFFERED PERMIT

REASONS FOR APPEAL OR OBJECTIONS: (Describe your reasons for appealing the decision or your objections to an initial proffered permit in clear concise statements. You may attach additional information to this form to clarify where your reasons or objections are addressed in the administrative record.)

ADDITIONAL INFORMATION: The appeal is limited to a review of the administrative record, the Corps memorandum for the record of the appeal conference or meeting, and any supplemental information that the review officer has determined is needed to clarify the administrative record. Neither the appellant nor the Corps may add new information or analyses to the record. However, you may provide additional information to clarify the location of information that is already in the administrative record.

POINT OF CONTACT FOR QUESTIONS OR INFORMATION:

If you have questions regarding this decision and/or the appeal process you may contact:
Alan Edmondson
Regulatory Branch
US Army Corps of Engineers
1222 Spruce Street
St. Louis, MO 63103-2833
Phone: (314)331-8186 Fax: (314)331-8741

If you only have questions regarding the appeal process you may also contact:
Martha S. Chieply, Administrative Appeals Review Officer
Mississippi Valley Division
P.O. Box 80
Vicksburg, MS 39181-0080
Phone: (601)634-5820 Fax: (601)634-5816

RIGHT OF ENTRY: Your signature below grants the right of entry to Corps of Engineers personnel, and any government consultants, to conduct investigations of the project site during the course of the appeal process. You will be provided a 15 day notice of any site investigation, and will have the opportunity to participate in all site investigations.

	Date:	Telephone number:
_____ Signature of appellant or agent.		

Nationwide Permit

U.S Army Corps
Of Engineers
St. Louis District

No. 6, Survey Activities
(NWP Final Notice, 67 FR 2079)

Survey activities including core sampling, seismic exploratory operations, plugging of seismic shot holes and other exploratory-type bore holes, soil survey and sampling, and historic resources surveys. Discharges and structures associated with the recovery of historic resources are not authorized by this NWP. Drilling and the discharge of excavated material from test wells for oil and gas exploration is not authorized by the NWP; the plugging of such wells is authorized. Fill placed for roads, pads and other similar activities is not authorized by the NWP. The NWP does not authorize any permanent structures. The discharge of drilling muds and cuttings may require a permit under Section 402 of the Clean Water Act. (Sections 10 and 404)

NATIONWIDE PERMIT CONDITIONS

General Conditions: The following general conditions must be followed in order for any authorization by a NWP to be valid:

1. **Navigation**. No activity may cause more than a minimal adverse effect on navigation.

2. **Proper Maintenance**. Any structure or fill authorized shall be properly maintained, including maintenance to ensure public safety.

3. **Soil Erosion and Sediment Controls**. Appropriate soil erosion and sediment controls must be used and maintained in effective operating condition during construction, and all exposed soil and other fills, as well as any work below the ordinary high water mark or high tide line, must be permanently stabilized at the earliest practicable date. Permittees are encouraged to perform work within waters of the United States during periods of low-flow or no-flow.

4. **Aquatic Life Movements**. No activity may substantially disrupt the life-cycle movements of those species of aquatic life indigenous to the waterbody, including those species that normally migrate through the area, unless the activity's primary purpose is to impound water. Culverts placed in streams must be installed to maintain low flow conditions.

5. **Equipment**. Heavy equipment working in wetlands must be placed on mats, or other measures must be taken to minimize soil disturbance.

6. **Regional and Case-By-Case Conditions**. The activity must comply with any regional conditions, which may have been added by the Division Engineer (see 33 CFR 330.4(e)) and with any case specific conditions added by the Corps or by the state or tribe in its Section 401 Water Quality Certification and Coastal Zone Management Act consistency determination.

7. **Wild and Scenic Rivers**. No activity may occur in a component of the National Wild and Scenic River System; or in a river officially designated by Congress as a "study river" for possible mix inclusion in the system, while the river is in an official study status; unless the appropriate Federal agency, with direct management responsibility for such river, has determined in writing that the proposed activity will not adversely affect the Wild and Scenic River designation, or study status. Information on Wild and Scenic Rivers may be obtained from the appropriate Federal land management agency in the area (e.g., National Park Service, US Forest Service, Bureau of Land Management, US Fish and Wildlife Service).

8. **Tribal Rights**. No activity or its operation may impair reserved tribal rights, including, but not limited to, reserved water rights and treaty fishing and hunting rights.

9. **Water Quality**. (a) In certain States and tribal lands an individual 401 water quality certification must be obtained or waived (See 33 CFR 330.4(c)).

(b) For NWPs 12, 14, 17, 18, 32, 39, 40, 42, 43, and 44, where the State or tribal 401 certification (either generically or individually) does not require or approve a water quality management measures, the permittee must provide water quality management measures that will ensure that the authorized work does not result in more than minimal degradation of water quality (or the Corps determines that compliance with state or local standards, where applicable, will ensure no more than minimal adverse effect on water quality). An important component of a water quality management plan includes stormwater management that minimizes degradation of the downstream aquatic system, including water quality (Refer to General Condition 21 for stormwater management requirements). Another important component of a water quality management plan is the establishment and maintenance of vegetated buffers next to open waters, including streams (Refer to General Condition 19 for vegetated buffer requirements for the NWPs). This condition is only applicable to projects that have the potential to affect water quality. While appropriate measures must be taken, in most cases it is not necessary to conduct detailed studies to identify such measures or to require monitoring.

10. **Coastal Zone Management**. In certain states, an individual state coastal zone management consistency concurrence must be obtained or waived (see 33 CFR 330.4(d)).

11. **Endangered Species**. (a) No activity is authorized under any NWP, which is likely to jeopardize the continued existence of a threatened or endangered species, or a species proposed for such designation, as identified under the Federal Endangered Species Act, or which will destroy or adversely modify the critical habitat of such species. Non-federal permittees shall notify the District Engineer if any listed species or designated critical habitat might be affected or is in the vicinity of the project, or is located in the designated critical habitat and shall not begin work on the activity until notified by the District Engineer that the requirements of the Endangered Species Act have been satisfied and that the activity is authorized. For activities that may affect Federally-listed endangered or threatened species or designated critical habitat, the notification must include the name(s) of the endangered or threatened species that may be affected by the proposed work or that utilize the

designated critical habitat that may be affected by the proposed work. As a result of formal or informal consultation with the FWS or NMFS, the District Engineer may add species-specific regional endangered species conditions to the NWPs.

(b) Authorization of an activity by a nationwide permit does not authorize the 'take' of a threatened or endangered species as defined under the Federal Endangered Species Act. In the absence of separate authorization (e.g., an ESA Section 10 Permit, a Biological Opinion with 'incidental take' provisions, etc.) from the US Fish and Wildlife Service or the National Marine Fisheries Service, both lethal and non-lethal 'takes' of protected species are in violation of the Endangered Species Act. Information on the location of threatened and endangered species and their critical habitat can be obtained directly from the offices of the US Fish and Wildlife Service and National Marine Fisheries Service or their World Wide Web pages at http://www.fws.gov/r9endspp/endspp.html and http://www.nfms.noaa.gov/prot_res/overview/es.html, respectively.

12. **Historic Properties**. No activity, which may affect historic properties, listed, or eligible for listing, in the National Register of Historic Places is authorized, until the DE has complied with the provisions of 33 CFR part 325, Appendix C. The prospective permittee must notify the District Engineer if the authorized activity may affect any historic properties listed, determined to be eligible, or which the prospective permittee has reason to believe may be eligible for listing on the National Register of Historic Places, and shall not begin the activity until notified by the District Engineer that the requirements of the National Historic Preservation Act have been satisfied and that the activity is authorized. Information on the location and existence of historic resources can be obtained from the State Historic Preservation Office and the National Register of Historic Places (see 33 CFR 330.4(g)). For activities that may affect historic properties listed in, or eligible for listing in, the National Register of Historic Places, the notification must state which historic property may be affected by the proposed work or include a vicinity map indicating the location of the historic property.

13. **Notification**. (a) Timing: Where required by the terms of the NWP, the prospective permittee must notify the District Engineer with a preconstruction notification (PCN) as early as possible. The District Engineer must determine if the PCN is complete within 30 days of the date of receipt and can request the additional information necessary to make the PCN complete only once. However, if the prospective permittee does not provide all of the requested information, then the District Engineer will notify the prospective permittee that the PCN is still incomplete and the PCN review process will not commence until all of the requested information has been received by the District Engineer. The prospective permittee shall not begin the activity:

(1) Until notified in writing by the District Engineer that the activity may proceed under the NWP with any special conditions imposed by the District or Division Engineer; or

(2) If notified in writing by the District or Division Engineer that an individual permit is required; or

(3) Unless 45 days have passed from the District Engineer's receipt of the complete notification and the prospective permittee has not received written notice from the District or Division Engineer. Subsequently, the permittee's right to proceed under the NWP may be modified, suspended, or revoked only in accordance with the procedure set forth in 33 CFR 330.5(d)(2).

(b) Contents of Notification: The notification must be in writing and include the following information:

(1) Name, address, and telephone numbers of the prospective permittee;

(2) Location of the proposed project;

(3) Brief description of the proposed project; the project's purpose; direct and indirect adverse environmental effects the project would cause; any other NWP(s), regional general permit(s), or individual permit(s) Used or intended to be Used to authorize any part of the proposed project or any related activity. Sketches should be provided when necessary to show that the activity complies with the terms of the NWP (Sketches usually clarify the project and when provided result in a quicker decision); and

(4) For NWPs 7, 12, 14, 18, 21, 34, 38, 39, 40, 41, 42, and 43, the PCN must also include a delineation of affected special aquatic sites, including wetlands, vegetated shallows (e.g., submerged aquatic vegetation, seagrass beds), and riffle and pool complexes (see paragraph 13(f));

(5) For NWP 7, Outfall Structures and Maintenance, the PCN must include information regarding the original design capacities and configurations of those areas of the facility where maintenance dredging or excavation is proposed.

(6) For NWP 14, Linear Transportation Projects, the PCN must include a compensatory mitigation proposal to offset permanent losses of waters of the US and a statement describing how temporary losses of waters of the US will be minimized to the maximum extent practicable.

(7) For NWP 21, Surface Coal Mining Activities, the PCN must include an Office of Surface Mining (OSM) or state-approved mitigation plan. To be authorized by this NWP, the District Engineer must determine that the activity complies with the terms and conditions of the NWP and that the adverse environmental effects are minimal both individually and cumulatively and must notify the project sponsor of this determination in writing;

(8) For NWP 27, Stream and Wetland Restoration Activities, the PCN must include documentation of the prior condition of the site that will be reverted by the permittee.

(9) For NWP 29, Single-Family Housing, the PCN must also include:

(i) Any past use of this NWP by the individual permittee and/or the permittee's spouse;

(ii) A statement that the single-family housing activity is for a personal residence of the permittee;

(iii) A description of the entire parcel, including its size, and a delineation of wetlands. For the purpose of this NWP, parcels of land measuring 1/4 acre or less will not require a formal on-site delineation. However, the applicant shall provide an indication of where the wetlands are and the amount of wetlands that exists on the property. For parcels greater than 1/4 acre in size, a formal wetland delineation must be prepared in accordance with the current method required by the Corps. (See paragraph 13(f));

(iv) A written description of all land (including, if available, legal descriptions) owned by the prospective permittee and/or the prospective permittee's spouse, within a one mile radius of the parcel, in any form of ownership (including any land owned as a partner, corporation, joint tenant, co-tenant, or as a tenant-by-the-entirety) and any land on which a purchase and sale agreement or other contract for sale or purchase has been executed;

(10) For NWP 31, Maintenance of Existing Flood Control Facilities, the prospective permittee must either notify the District Engineer with a PCN prior to each maintenance activity or submit a five year (or less) maintenance plan. In addition, the PCN must include all of the following:

(i) Sufficient baseline information so as to identify the approved channel depths and configurations and existing facilities. Minor deviations are authorized, provided the approved flood control protection or drainage is not increased;

(ii) A delineation of any affected special aquatic sites, including wetlands; and,

(iii) Location of the dredged material disposal site.

(11) For NWP 33, Temporary Construction, Access, and Dewatering, the PCN must also include a restoration plan of reasonable measures to avoid and minimize adverse effects to aquatic resources.

(12) For NWP's 39, 43, and 44, the PCN must also include a written statement to the District Engineer explaining how avoidance and minimization of losses of waters of the US were achieved on the project site.

(13) For NWP 39 and NWP 42, the PCN must include a compensatory mitigation proposal that offsets unavoidable losses of waters of the US or justification explaining why compensatory mitigation should not be required. For discharges that cause the loss of greater than 300 linear feet of an intermittent stream bed, to be authorized, the District Engineer must determine that the activity complies with the other terms and conditions of the NWP, determine adverse environmental effects are minimal both individually and cumulatively, and waive the limitation on stream impacts in writing before the permitttee may proceed;

(14) For NWP 40 (Agricultural Activities), the PCN must include a compensatory mitigation proposal to offset losses of waters of the US. This NWP does not authorize the relocation of greater than 300 linear-feet of existing serviceable drainage ditches constructed in non-tidal streams unless, for drainage ditches constructed in intermittent non-tidal streams, the District Engineer waives this criterion in writing, and the District Engineer has determined that the project complies with all terms and conditions of this NWP, and that any adverse impacts of the project on the aquatic environment are minimal, both individually and cumulatively;

(15) For NWP 43 (Stormwater Management Facilities), the PCN must include, for the construction of new stormwater management facilities, a maintenance plan (in accordance with state and local requirements, if applicable) and a compensatory mitigation proposal to offset losses of waters of the US. For discharges that cause the loss of greater than 300 linear feet of an intermittent streambed, to be authorized, the District Engineer must determine that the activity complies with the other terms and conditions of the NWP, determine adverse environmental effects are minimal both individually and cumulatively, and waive the limitation on stream impacts in writing before the permittee may proceed;

(16) For NWP 44, Mining Activities, the PCN must include a description of all waters of the US adversely affected by the project, a description of measures taken to minimize adverse effects to waters of the US, a description of measures taken to comply with the criteria of the NWP, and a reclamation plan (for aggregate mining activities in isolated waters and non-tidal wetlands adjacent to headwaters and any hard rock/mineral mining activities).

(17) For activities that may adversely affect Federally-listed endangered or threatened species, the PCN must include the name(s) of those endangered or threatened species that may be affected by the proposed work or utilize the designated critical habitat that may be affected by the proposed work.

(18) For activities that may affect historic properties listed in, or eligible for listing in, the National Register of Historic Places, the PCN must state which historic property may be affected by the proposed work or include vicinity map indicating the location of the historic property.

(c) Form of Notification: The standard individual permit application form (Form ENG 4345) may be Used as the notification but must clearly indicate that it is a PCN and must include all of the information required in (b) (1)-(19) of General Condition 13. A letter containing the requisite information may also be used.

(d) District Engineer's Decision: In reviewing the PCN for the proposed activity, the District Engineer will determine whether the activity authorized by the NWP will result in more than minimal individual or cumulative adverse environmental effects or may be contrary to the public interest. The prospective permittee may, optionally, submit a proposed mitigation plan with the PCN to expedite the process and the District Engineer will consider any proposed compensatory mitigation the applicant has included in the proposal in determining whether the net adverse environmental effects to the aquatic environment of the proposed work are minimal. If the District Engineer determines that the activity complies with the terms and conditions of the NWP and that the adverse effects on the aquatic environment are minimal, the District Engineer will notify the permittee and include any conditions the District Engineer deems necessary. Any compensatory mitigation proposal must be approved by the District Engineer prior to commencing work. If the prospective permittee is required to submit a compensatory mitigation proposal with the PCN, the proposal may be either conceptual or detailed. If the prospective permittee elects to submit a compensatory mitigation plan with the PCN, the District Engineer will expeditiously review the proposed compensatory mitigation plan. The District Engineer must review the plan within 45 days of receiving a complete PCN and determine whether the conceptual or specific proposed mitigation would ensure no more than minimal adverse effects on the aquatic environment. If the net adverse effects of the project on the aquatic environment (after consideration of the compensatory mitigation proposal) are determined by the District Engineer to be minimal, the District Engineer will provide a timely written response to the applicant stating that the project can proceed under the terms and conditions of the nationwide permit. If the District Engineer determines that the adverse effects of the proposed work are more than minimal, then he will notify the applicant either: (1) That the project does not qualify for authorization under the NWP and instruct the applicant on the procedures to seek authorization under an individual permit; (2) that the project is authorized under the NWP subject to the applicant's submission of a mitigation proposal that would reduce the adverse effects on the aquatic environment to the minimal level; or (3) that the project is authorized under the NWP with specific modifications or conditions. Where the District Engineer determines that mitigation is required in order to ensure no more than minimal adverse effects on the aquatic environment, the activity will be authorized within the 45-day PCN period, including the necessary conceptual or specific mitigation or a requirement that the applicant submit a mitigation proposal that would reduce the adverse effects on the aquatic environment to the minimal level. When conceptual mitigation is included, or a mitigation plan is required under item (2) above, no work in waters of the US will occur until the District Engineer has approved a specific mitigation plan.

(e) Agency Coordination: The District Engineer will consider any comments from Federal and State agencies concerning the proposed activity's compliance with the terms and conditions of the NWPs and the need for mitigation to reduce the project's adverse effects on the aquatic environment to a minimal level.

For activities requiring notification to the District Engineer that result in

the loss of greater than 1/2 acre of waters of the US, the District Engineer will, upon receipt of a notification, provide immediately (e.g., via facsimile transmission, overnight mail, or other expeditious manner), a copy to the appropriate offices of the Fish and Wildlife Service, State natural resource water quality agency, EPA, State Historic Preservation Officer (SHPO), and, if appropriate, the National Marine Fisheries Service. With the exception of NWP 37, these agencies will then have 10 calendar days from the date the material is transmitted to telephone or fax the District Engineer notice that they intend to provide substantive, site-specific comments. If so contacted by an agency, the District Engineer will wait an additional 15 calendar days before making a decision on the notification. The District Engineer will fully consider agency comments received within the specified time frame, but will provide no response to the resource agency, except as provided below. The District Engineer will indicate in the administrative record associated with each notification that the resource agencies' concerns were considered. As required by Section 305(b)(4)(B) of the Magnuson-Stevens Fishery Conservation and Management Act, the District Engineer will provide a response to National Marine Fisheries Service within 30 days of receipt of any Essential Fish Habitat conservation recommendations. Applicants are encouraged to provide the Corps multiple copies of notifications to expedite agency notification.

(f) Wetlands Delineations: Wetland delineations must be prepared in accordance with the current method required by the Corps. For NWP 29 see paragraph (b)(9)(iii) for parcels less than 1/4 acre in size. The permittee may ask the Corps to delineate the special aquatic site. There may be some delay if the Corps does the delineation. Furthermore, the 45-day period will not start until the wetland delineation has been completed and submitted to the Corps, where appropriate.

14. **Compliance Certification**. Every permittee who has received a nationwide permit verification from the Corps will submit a signed certification regarding the completed work and any required mitigation. The certification will be forwarded by the Corps with the authorization letter. The certification will include: (a) A statement that the authorized work was done in accordance with the Corps authorization, including any general or specific conditions; (b) A statement that any required mitigation was completed in accordance with the permit conditions; and (c) The signature of the permittee certifying the completion of the work and mitigation.

15. **Use of Multiple Nationwide Permits**. The use of more than one NWP for a single and complete project is prohibited, except when the acreage loss of waters of the US authorized by the NWPs does not exceed the acreage limit of the NWP with the highest specified acreage limit. For example, if a road crossing over tidal waters is constructed under NWP 14, with associated bank stabilization authorized by NWP 13, the maximum acreage loss of waters of the US for the total project cannot exceed 1/3 acre.

16. **Water Supply Intakes**. No activity, including structures and work in navigable waters of the US or discharges of dredged or fill material, may occur in the proximity of a public water supply intake except where the activity is for repair of the public water supply intake structures or adjacent bank stabilization.

Shellfish Beds. No activity, including structures and work in navigable waters of the US or discharges of dredged or fill material, may occur in areas of concentrated shellfish populations, unless the activity is directly related to a shellfish harvesting activity authorized by NWP 4.

18. **Suitable Material**. No activity, including structures and work in navigable waters of the US or discharges of dredged or fill material, may consist of unsuitable material (e.g., trash, debris, car bodies, asphalt, etc.) and material Used for construction or discharged must be free from toxic pollutants in toxic amounts (see Section 307 of the Clean Water Act).

19. Mitigation. The District Engineer will consider the factors discussed below when determining the acceptability of appropriate and practicable mitigation necessary to offset adverse effects on the aquatic environment that are more than minimal.

(a) The project must be designed and constructed to avoid and minimize adverse effects to waters of the US to the maximum extent practicable at the project site (i.e., on site).

(b) Mitigation in all its forms (avoiding, minimizing, rectifying, reducing or compensating) will be required to the extent necessary to ensure that the adverse effects to the aquatic environment are minimal.

(c) Compensatory mitigation at a minimum one-for-one ratio will be required for all wetland impacts requiring a PCN, unless the District Engineer determines in writing that some other form of mitigation would be more environmentally appropriate and provides a project-specific waiver of this requirement. Consistent with National policy, the District Engineer will establish a preference for restoration of wetlands as compensatory mitigation, with preservation used only in exceptional circumstances.

(d) Compensatory mitigation (i.e., replacement or substitution of aquatic resources for those impacted) will not be used to increase the acreage losses allowed by the acreage limits of some of the NWPs. For example, 1 /4 -acre of wetlands cannot be created to change a 3 /4 -acre loss of wetlands to a 1 /2 -acre loss associated with NWP 39 verification. However, 1 /2 -acre of created wetlands can be used to reduce the impacts of a 1 /2 -acre loss of wetlands to the minimum impact level in order to meet the minimal impact requirement associated with NWPs.

(e) To be practicable, the mitigation must be available and capable of being done considering costs, existing technology, and logistics in light of the overall project purposes. Examples of mitigation that may be appropriate and practicable include, but are not limited to: reducing the size of the project; establishing and maintaining wetland or upland vegetated buffers to protect open waters such as streams; and replacing losses of aquatic resource functions and values by creating, restoring, enhancing, or preserving similar functions and values, preferably in the same watershed.

(f) Compensatory mitigation plans for projects in or near streams or other open waters will normally include a requirement for the establishment, maintenance, and legal protection (e.g., easements, deed restrictions) of vegetated buffers to open waters. In many cases, vegetated buffers will be the only compensatory mitigation required. Vegetated buffers should consist of native species. The width of the vegetated buffers required will address documented water quality or aquatic habitat loss concerns. Normally, the vegetated buffer will be 25 to 50 feet wide on each side of the stream, but the District Engineers may require slightly wider vegetated buffers to address documented water quality or habitat loss concerns. Where both wetlands and open waters exist on the project site, the Corps will determine the appropriate compensatory mitigation (e.g., stream buffers or wetlands compensation) based on what is best for the aquatic environment on a watershed basis. In cases where vegetated buffers are determined to be the most appropriate form of compensatory mitigation, the District Engineer may waive or reduce the requirement to provide wetland compensatory mitigation for wetland impacts.

(g) Compensatory mitigation proposals submitted with the "notification" may be either conceptual or detailed. If conceptual plans are approved under the verification, then the Corps will condition the

verification to require detailed plans be submitted and approved by the Corps prior to construction of the authorized activity in waters of the US.

(h) Permittees may propose the use of mitigation banks, in-lieu fee arrangements or separate activity-specific compensatory mitigation. In all cases that require compensatory mitigation, the mitigation provisions will specify the party responsible for accomplishing and/or complying with the mitigation plan.

20. **Spawning Areas**. Activities, including structures and work in navigable waters of the US or discharges of dredged or fill material, in spawning areas during spawning seasons must be avoided to the maximum extent practicable. Activities that result in the physical destruction (e.g., excavate, fill, or smother downstream by substantial turbidity) of an important spawning area are not authorized.

21. **Management of Water Flows**. To the maximum extent practicable, the activity must be designed to maintain preconstruction downstream flow conditions (e.g., location, capacity, and flow rates). Furthermore, the activity must not permanently restrict or impede the passage of normal or expected high flows (unless the primary purpose of the fill is to impound waters) and the structure or discharge of dredged or fill material must withstand expected high flows. The activity must, to the maximum extent practicable, provide for retaining excess flows from the site, provide for maintaining surface flow rates from the site similar to preconstruction conditions, and provide for not increasing water flows from the project site, relocating water, or redirecting water flow beyond preconstruction conditions. Stream channelizing will be reduced to the minimal amount necessary, and the activity must, to the maximum extent practicable, reduce adverse effects such as flooding or erosion downstream and upstream of the project site, unless the activity is part of a larger system designed to manage water flows. In most cases, it will not be a requirement to conduct detailed studies and monitoring of water flow.

This condition is only applicable to projects that have the potential to affect waterflows. While appropriate measures must be taken, it is not necessary to conduct detailed studies to identify such measures or require monitoring to ensure their effectiveness. Normally, the Corps will defer to state and local authorities regarding management of water flow.

22. **Adverse Effects From Impoundments**. If the activity, including structures and work in navigable waters of the US or discharge of dredged or fill material, creates an impoundment of water, adverse effects on the aquatic system caused by the accelerated passage of water and/or the restriction of its flow shall be minimized to the maximum extent practicable.

23. **Waterfowl Breeding Areas**. Activities, including structures and work in navigable waters of the US or discharges of dredged or fill material, into breeding areas for migratory waterfowl must be avoided to the maximum extent practicable.

24. **Removal of Temporary Fills**. Any temporary fills must be removed in their entirety and the affected areas returned to their preexisting elevation.

25. **Designated Critical Resource Waters**. Critical resource waters include, NOAA-designated marine sanctuaries, National Estuarine Research Reserves, National Wild and Scenic Rivers, critical habitat for Federally listed threatened and endangered species, coral reefs, State natural heritage sites, and outstanding national resource waters or other waters officially designated by a State as having particular environmental or ecological significance and identified by the District Engineer after notice and opportunity for public comment. The District Engineer may also designate additional critical resource waters after notice and opportunity for comment.

(a) Except as noted below, discharges of dredged or fill material into waters of the US are not authorized by NWPs 7, 12, 14, 16, 17, 21, 29, 31, 35, 39, 40, 42, 43, and 44 for any activity within, or directly affecting, critical resource waters, including wetlands adjacent to such waters. Discharges of dredged or fill materials into waters of the US may be authorized by the above NWPs in National Wild and Scenic Rivers if the activity complies with General Condition 7. Further, such discharges may be authorized in designated critical habitat for Federally listed threatened or endangered species if the activity complies with General Condition 11 and the US Fish and Wildlife Service or the National Marine Fisheries Service has concurred in a determination of compliance with this condition.

(b) For NWPs 3, 8, 10, 13, 15, 18, 19, 22, 23, 25, 27, 28, 30, 33, 34, 36, 37, and 38, notification is required in accordance with General Condition 13, for any activity proposed in the designated critical resource waters including wetlands adjacent to those waters. The District Engineer may authorize activities under these NWPs only after he determines that the impacts to the critical resource waters will be no more than minimal.

26. **Fills Within 100-Year Floodplains**. For purposes of this General Condition, 100-year floodplains will be identified through the existing Federal Emergency Management Agency's (FEMA) Flood Insurance Rate Maps or FEMA-approved local floodplain maps.

(a) *Discharges in Floodplain; Below Headwaters*. Discharges of dredged or fill material into waters of the US within the mapped 100-year floodplain, below headwaters (i.e. five cfs), resulting in permanent above-grade fills, are not authorized by NWPs 39, 40, 42, 43, and 44.

(b) *Discharges in Floodway; Above Headwaters*. Discharges of dredged or fill material into waters of the US within the FEMA or locally mapped floodway, resulting in permanent above-grade fills, are not authorized by NWPs 39, 40, 42, and 44.

(c) The permittee must comply with any applicable FEMA-approved state or local floodplain management requirements.

27. Construction Period. For activities the Corps has not verified that and the project were commenced or under contract to commence by the expiration date of the NWP (or modification or revocation date), the work must be completed within 12- months after such date (including any modification that affects the project).

For activities that have been verified and the project was commenced or under contract to commence within the verification period, the work must be completed by the date determined by the Corps.

For projects that have been verified by the Corps, an extension of a Corps approved completion date maybe requested. This request must be submitted at least one month before the previously approved completion date.

D. Further Information

1. District engineers have authority to determine if an activity complies with the terms and conditions of an NWP.

2. NWPs do not obviate the need to obtain other Federal, State, or local permits, approvals, or authorizations required by law.

3. NWPs do not grant any property rights or exclusive privileges.

4. NWPs do not authorize any injury to the property or rights of others.

5. NWPs do not authorize interference with any existing or proposed Federal project.

Section 10 Special Condition: The permittee understands and agrees that, if future operations by the US require the removal, relocation, or other

alteration, of the structure or work herein authorized, or if, in the opinion of the Secretary of the Army or is authorized representative, said structure or work shall cause unreasonable obstruction to the free navigation of the navigable waters, the permittee will be required, upon due notice from the Corps of Engineers, to remove, relocate, or alter the structure work or obstructions caused thereby, without expense to the US. No claim shall be made against the US on account of any such removal or alteration.

DEFINITIONS

Best management practices: Best Management Practices (BMPs) are policies, practices, procedures, or structures implemented to mitigate the adverse environmental effects on surface water quality resulting from development. BMPs are categorized as structural or non-structural. A BMP policy may affect the limits on a development.

Compensatory mitigation: For purposes of Section 10/404, compensatory mitigation is the restoration, creation, enhancement, or in exceptional circumstances, preservation of wetlands and/or other aquatic resources for the purpose of compensating for unavoidable adverse impacts, which remain, after all appropriate and practicable avoidance and minimization has been achieved.

Creation: The establishment of a wetland or other aquatic resource where one did not formerly exist.

Enhancement: Activities conducted in existing wetlands or other aquatic resources, which increase one or more aquatic functions.

Ephemeral stream: An ephemeral stream has flowing water only during, and for a short duration after, precipitation events in a typical year. Ephemeral streambeds are located above the water table year-round. Groundwater is not a source of water for the stream. Runoff from rainfall is the primary source of water for stream flow.

Farm tract: A unit of contiguous land under one ownership which is operated as a farm or part of a farm.

Flood Fringe: That portion of the 100-year floodplain outside of the floodway (often referred to as ``floodway fringe."

Floodway: The area regulated by Federal, state, or local requirements to provide for the discharge of the base flood so the cumulative increase in water surface elevation is no more than a designated amount (not to exceed one foot as set by the National Flood Insurance Program) within the 100-year floodplain.

Independent utility: A test to determine what constitutes a single and complete project in the Corps regulatory program. A project is considered to have independent utility if it would be constructed absent the construction of other projects in the project area. Portions of a multi-phase project that depend upon other phases of the project do not have independent utility. Phases of a project that would be constructed even if the other phases are not built can be considered as separate single and complete projects with independent utility.

Intermittent stream: An intermittent stream has flowing water during certain times of the year, when groundwater provides water for stream flow. During dry periods, intermittent streams may not have flowing water. Runoff from rainfall is a supplemental source of water for stream flow.

Loss of waters of the US: Waters of the US that include the filled area and other waters that are permanently adversely affected by flooding, excavation, or drainage because of the regulated activity. Permanent adverse effects include permanent above-grade, at-grade, or below-grade fills that change an aquatic area to dry land, increase the bottom elevation of a waterbody, or change the use of a waterbody. The acreage of loss of waters of the US is the threshold measurement of the impact to existing waters for determining whether a project may qualify for an NWP; it is not a net threshold that is calculated after considering compensatory mitigation that may be used to offset losses of aquatic functions and values. The loss of stream bed includes the linear feet of stream bed that is filled or excavated. Waters of the US temporarily filled, flooded, excavated, or drained, but restored to preconstruction contours and elevations after construction, are not included in the measurement of loss of waters of the US. Impacts to ephemeral waters are only not included in the acreage or linear foot measurements of loss of waters of the US or loss of stream bed, for the purpose of determining compliance with the threshold limits of the NWPs.

Non-tidal wetland: A non-tidal wetland is a wetland (i.e., a water of the US) that is not subject to the ebb and flow of tidal waters. The definition of a wetland can be found at 33 CFR 328.3(b). Non-tidal wetlands contiguous to tidal waters are located landward of the high tide line (i.e., the spring high tide line).

Open water: An area that, during a year with normal patterns of precipitation, has standing or flowing water for sufficient duration to establish an ordinary high water mark. Aquatic vegetation within the area of standing or flowing water is either non-emergent, sparse, or absent. Vegetated shallows are considered to be open waters. The term `open water' includes rivers, streams, lakes, and ponds. For the purposes of the NWPs, this term does not include ephemeral waters.

Perennial stream: A perennial stream has flowing water year-round during a typical year. The water table is located above the streambed for most of the year. Groundwater is the primary source of water for stream flow. Runoff from rainfall is a supplemental source of water for stream flow.

Permanent above-grade fill: A discharge of dredged or fill material into waters of the US, including wetlands, that results in a substantial increase in ground elevation and permanently converts part or all of the waterbody to dry land. Structural fills authorized by NWPs 3, 25, 36, etc. are not included.

Preservation: The protection of ecologically important wetlands or other aquatic resources in perpetuity through the implementation of appropriate legal and physical mechanisms. Preservation may include protection of upland areas adjacent to wetlands as necessary to ensure protection and/or enhancement of the overall aquatic ecosystem.

Restoration: Re-establishment of wetland and/or other aquatic resource characteristics and function(s) at a site where they have ceased to exist, or exist in a substantially degraded state

Riffle and pool complex: Riffle and pool complexes are special aquatic sites under the 404(b)(1) Guidelines. Riffle and pool complexes sometimes characterize steep gradient sections of streams. Such stream sections are recognizable by their hydraulic characteristics. The rapid movement of water over a course substrate in riffles results in a rough flow, turbulent surface, and high dissolved oxygen levels in the water. Pools are deeper areas associated with riffles. Pools are characterized by a slower stream velocity, a streaming flow, a smooth surface, and a finer substrate.

Single and complete project: The term `single and complete project' is defined at 33 CFR 330.2(i) as the total project proposed or accomplished by one owner/developer or partnership or other association of owners/developers (see definition of independent utility). For linear projects, the `single and complete project' (i.e., a single and complete crossing) will apply to each crossing of a separate water of the US (i.e., a single waterbody) at that location. An exception is for linear projects crossing a single waterbody several times at separate and distant

locations: each crossing is considered a single and complete project. However, individual channels in a braided stream or river, or individual arms of a large, irregularly shaped wetland or lake, etc., are not separate waterbodies.

Stormwater management: Stormwater management is the mechanism for controlling stormwater runoff for the purposes of reducing downstream erosion, water quality degradation, and flooding and mitigating the adverse effects of changes in land use on the aquatic environment.

Stormwater management facilities: Stormwater management facilities are those facilities, including but not limited to, stormwater retention and detention ponds and BMPs, which retain water for a period of time to control runoff and/or improve the quality (i.e., by reducing the concentration of nutrients, sediments, hazardous substances and other pollutants) of stormwater runoff.

Streambed: The substrate of the stream channel between the ordinary high water marks. The substrate may be bedrock or inorganic particles that range in size from clay to boulders. Wetlands contiguous to the streambed, but outside of the ordinary high water marks, are not considered part of the streambed.

Stream channelization: The manipulation of a stream channel to increase the rate of water flow through the stream channel. Manipulation may include deepening, widening, straightening, armoring, or other activities that change the stream cross-section or other aspects of stream channel geometry to increase the rate of water flow through the stream channel. A channelized stream remains a water of the US, despite the modifications to increase the rate of water flow.

Tidal wetland: A tidal wetland is a wetland (i.e., a water of the US) that is inundated by tidal waters. The definitions of a wetland and tidal waters can be found at 33 CFR 328.3(b) and 33 CFR 328.3(f), respectively. Tidal waters rise and fall in a predictable and measurable rhythm or cycle due to the gravitational pulls of the moon and sun. Tidal waters end where the rise and fall of the water surface can no longer be practically measured in a predictable rhythm due to masking by other waters, wind, or other effects. Tidal wetlands are located channelward of the high tide line (i.e., spring high tide line) and are inundated by tidal waters two times per lunar month, during spring high tides.

Vegetated buffer: A vegetated upland or wetland area next to rivers, streams, lakes, or other open waters, which separates the open water from developed areas, including agricultural land. Vegetated buffers provide a variety of aquatic habitat functions and values (e.g., aquatic habitat for fish and other aquatic organisms, moderation of water temperature changes, and detritus for aquatic food webs) and help improve or maintain local water quality. A vegetated buffer can be established by maintaining an existing vegetated area or planting native trees, shrubs, and herbaceous plants on land next to open waters. Mowed lawns are not considered vegetated buffers because they provide little or no aquatic habitat functions and values. The establishment and maintenance of vegetated buffers is a method of compensatory mitigation that can be used in conjunction with the restoration, creation, enhancement, or preservation of aquatic habitats to ensure that activities authorized by NWPs result in minimal adverse effects to the aquatic environment. (See General Condition 19.)

Vegetated shallow: Vegetated shallows are special aquatic sites under the 404(b)(1) Guidelines. They are areas that are permanently inundated and under normal circumstances have rooted aquatic vegetation, such as seagrasses in marine and estuarine systems and a variety of vascular rooted plants in freshwater systems.

Waterbody: A waterbody is any area that in a normal year has water flowing or standing above ground to the extent that evidence of an ordinary high water mark is established. Wetlands contiguous to the waterbody are considered part of the waterbody.

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September 16th, 2005

Mr. Kenneth Bardo
U.S. EPA Region V
Corrective Action Section
Enforcement Compliance Branch
77 West Jackson Boulevard DE-J9
Chicago, IL 60604-3507

Re: Solutia Inc. – W. G. Krummrich Plant, RCRA
PCB Mobility and Migration Investigation Plan

Dear Ken:

Enclosed please find our proposed PCB Mobility and Migration Investigation Plant for the W. G. Krummrich Plant.

We look forward to discussing this document with you in the near future.

Sincerely,

Steven D. Smith
Project Manager

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Background - PCBs are present in unsaturated soil in the Former PCB Manufacturing Area at the W.G. Krummrich Facility with concentrations ranging from non-detect (< 110 ug/kg) to 22,100 mg/kg (Figure 1):

Summary of Maximum Detected Total PCB Concentrations in Former PCB Manufacturing Area Soils

Soil Boring	Maximum Detected PCB Concentration (mg/kg)	Soil Boring	Maximum Detected PCB Concentration (mg/kg)
S0801	ND	S0830	1,470
S0803	8	S0831	2,030
S0836	10.6	S0813	2,200
S0834	85.0	S0822	2,207
S0827	98.0	S0802	2,570
S0806	154	S0826	3,130
S0819	430	S0810	3,290
S0823	930	S0835	9,300
S0804	1,140	S0825	22,100

For this data set, the average maximum detected PCB concentration is 3009 mg/kg.

In September 1984, Aroclor 1254 was detected at a concentration of 4.99 ug/l in Monitoring Well GM-15. Monitoring Well GM-15 is located on the north side of the Former PCB Manufacturing Area and is screened from 15 to 38 feet below ground surface (ft bgs). This monitoring well, which was only sampled once, is completed in the Shallow Hydrogeologic Unit (SHU):

Monitoring Well GM-15 Boring Log

Soil Type	Description	Depth Below Ground Surface (Feet)
Fill	Gravel	0 to 2
Silt	Clayey, Gray and Brown; Some Sand	2 to 9
Sand	Very Fine to Fine, Silty, Gray	9 to 38

Total VOCs and Total SVOCs were also detected in the September 1984 sample collected from GM-15 at concentrations of 153 ug/l and 245 ug/l, respectively. The constituents that comprised the Total VOCs and Total SVOCs are listed below:

Summary of Volatile and Semivolatile Organic Compounds Detected in Monitoring Well GM-15

Volatile Organic Compounds, ug/l		Semivolatile Organic Compounds, ug/l	
Benzene	8	1,2-Dichlorobenzene	185
Chlorobenzene	103	1,3-Dichlorobenzene	35
Ethylbenzene	9	Diethyl Phthalate	1
Methylene Chloride	24	Di-n-butyl Phthalate	3
Toluene	9	Nitrobenzene	8
		Pentachlorophenol	1
Total VOCs	153	1,24-Trichlorophenol	12
		Total SVOCs	245

Objective - The objective of the PCB Mobility and Migration Investigation is to determine whether or not

PCBs in unsaturated soils at the Former PCB Manufacturing Area migrate to underlying and downgradient groundwater. PCBs were manufactured at this unit from the 1930s to 1977, when production ceased and the unit was dismantled. Spills and leaks over more than 40 years of operation resulted in PCB concentrations in unsaturated soils that range from non-detect to 22,100 mg/kg. A September 1984 sample indicates 4.99 ug/l of Aroclor 1254 were present in groundwater 15 to 38 ft bgs on the northern side of the Former PCB Manufacturing Area. It is likely that this sample, collected 21 years ago, was not filtered, and that the PCB concentration in groundwater water due to PCBs bound to suspended solids entrained during groundwater sample collection. Monitoring Well GM-15 was screened in silt and very fine, silty sand. Collection of soil and groundwater samples in this area will allow evaluation of PCB leaching from unsaturated soils and migration to underlying and downgradient groundwater.

Site Investigation - Soil samples will be collected in five foot increments from ground surface to15 ft bgs using push-sampling methods to characterize unsaturated zone soils in the Former PCB Manufacturing Area. Samples will be collected at the three previous soil sampling locations with the highest maximum detected PCB concentrations. Specifically, soil samples will be collected at sampling location S0825 (22,100 mg/kg), S0835 (9,300 mg/kg) and S0810 (3,290 mg/kg) from depths of 0 to 5, 5 to 10 and 10 to 15 ft bgs (Figure 1). These soil sampling locations will be designated PMA-S-1 (S0825), PMA-S-2 (S0835) and PMA-S-3 (S0810). Samples will be analyzed for Total and SPLP-extractable VOCs, SVOCs and PCBs using USEPA Methods 8260B, 8270C and 680, respectively. USEPA Method 1312, Synthetic Precipitation Leaching Procedure, will be used to create an extract that can be used to evaluate constituent mobility after analysis by Methods 8260B (VOCs), 8270C (SVOCs) and 680 (PCBs). Dichlorobenzenes will be quantitated using Method 8260B because of volatilization losses associated with Method 8270C. VOC samples will be selected for analysis based on the highest PID reading within each 5 ft sample interval and collected using Method 5035. If there is evidence of staining or other visual indicators of waste materials, the stained portion of each 5 ft sample interval will be selected for SVOC and PCB analysis. If there is no evidence of staining, each 5 ft sample interval will be thoroughly homogenized before sampling. A total of 18 soil samples will be collected. Nine soil samples will be analyzed for Total VOCs, SVOCs and PCBs and nine will be analyzed for SPLP-extractable VOCs, SVOCs and PCBs.

Groundwater samples will be collected from depths of 20, 40 and 60 ft bgs at soil sampling locations PMA-S-1, PMA-S-2 and PMA-S-3 using push sampling methods to determine if PCBs are migrating from the unsaturated zone to the saturated zone. These groundwater sampling locations will be designated PMA-GW-1 (S0825), PMA-GW-2 (S0835) and PMA-GW-3 (S0810). Unfiltered and filtered samples will be analyzed for VOCs (Method 8260B), SVOCs (Method 8270C) and PCBs (Method 680). A total of 18 groundwater samples will be collected. Nine groundwater samples will be analyzed for unfiltered VOCs,

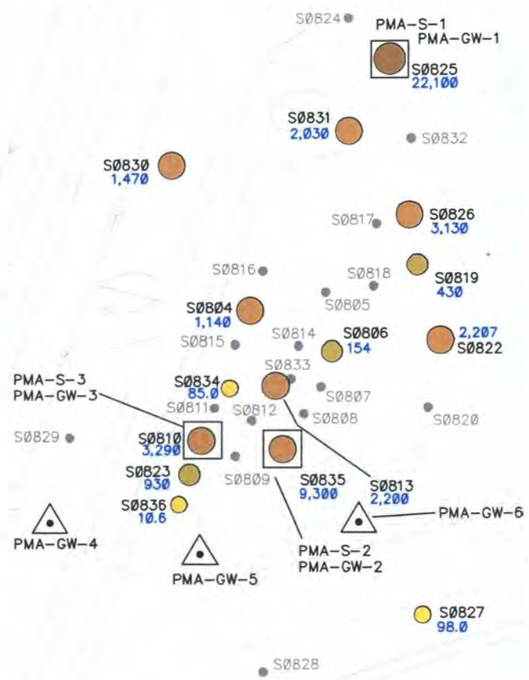
SVOCs and PCBs and nine will be analyzed for filtered VOCs, SVOCs and PCBs.

Groundwater samples will be collected from depths of 20, 40 and 60 ft bgs using push-sampling methods at three locations on the downgradient limit of the Former PCB Manufacturing Area to determine if PCBs are migrating downgradient of this potential source area (Figure 1). These groundwater sampling locations will be designated PMA-GW-4, PMA-GW-5 and PMA-GW-6. Unfiltered and filtered samples will be analyzed for VOCs (Method 8260B), SVOCs (Method 8270C) and PCBs (Method 680). A total of 18 groundwater samples will be collected. Nine groundwater samples will be analyzed for unfiltered VOCs, SVOCs and PCBs and nine will be analyzed for filtered VOCs, SVOCs and PCBs.

Implementation - Existing Field Sampling Plans, Quality Assurance Project Plans and Health and Safety Plans will be used to perform this work.

Report - On completion of data validation, a report will be prepared summarizing collected soil and groundwater data, evaluating whether or not PCBs are mobilized from the unsaturated to the saturated zone and determining whether or not PCBs are migrating downgradient from this potential source area.

Schedule - Barring unforeseen difficulties with equipment and/or labor availability and unanticipated problems with laboratory turnaround times, sample collection, laboratory analysis, data validation and report preparation should be completed in 90 to 120 days.



LEGEND

RESULTS (in mg/kg)

- ND (NON DETECT)
- < 1
- 1 – 10
- 10 – 100
- 100 – 1,000
- 1,000 – 10,000
- > 10,000
- BORING LOCATIONS
- SOIL AND GROUNDWATER SAMPLING LOCATIONS
- GROUNDWATER SAMPLING LOCATIONS

PMA = FORMER PCB MANUFACTURING AREA

S = SOIL

GW = GROUNDWATER

NOTES:

1) DATA DEPICTED ON THIS FIGURE WERE COLLECTED DURING TWO SEPARATE CMS SOIL SAMPLING EVENTS.

2) THE DOTS SHOWN ON THE FIGURE REPRESENT THE HIGHEST TOTAL CONCENTRATION (SUMATION OF ALL CONSTITUENTS). HIGHEST CONCENTRATIONS ARE SHOWN IN BLUE FONT.

3) THE DEPTH INTERVALS ARE AS FOLLOWS:
SHALLOW = 0–3FT BGS
INTERMEDIATE = 4–12FT BGS
DEEP = 13–16FT BGS

4) FIELD SCREENING WAS PERFORMED ON ALL BORING LOCATIONS. BASED ON SCREENING RESULTS, SAMPLES WERE SUBMITTED FOR LABORATORY ANALYSIS.

N E S W

0 50

SCALE FEET

PCB MOBILITY AND MIGRATION INVESTIGATION W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS	PROJECT NO. 21561600.00000

URS

DRN. BY: 7/14/04 DSGN. BY: kef CHKD. BY:	SAMPLE LOCATIONS	FIG. NO. 1

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September 16th, 2005

Mr. Kenneth Bardo
U.S. EPA Region V
Corrective Action Section
Enforcement Compliance Branch
77 West Jackson Boulevard DE-J9
Chicago, IL 60604-3507

Re: Solutia Inc. – W. G. Krummrich Plant, RCRA
Plume Stability Monitoring Plan

Dear Ken:

Enclosed is the revised, Plume Stability Monitoring Plan for the W. G. Krummrich Plant.

We look forward to discussing this document with you in the near future.

Sincerely,

Steven D. Smith
Project Manager

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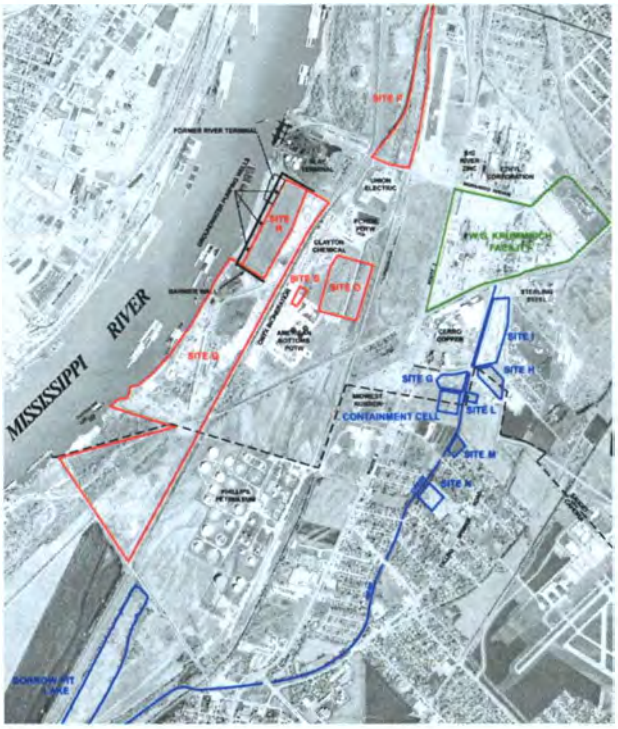
SOLUTIA-209

PLUME STABILITY MONITORING PLAN

SOLUTIA INC.
W.G. KRUMMRICH FACILITY
SAUGET, ILLINOIS



Prepared by:
Solutia Inc.
575 Maryville Centre Dr.
St. Louis, Missouri 63141

SOLUTIA

URS

September 16, 2005

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1.0 INTRODUCTION

On May 3, 2000, USEPA executed a Resource Conservation and Recovery Act (RCRA) 3008(h) Administrative Order on Consent for Solutia Inc.'s W.G. Krummrich facility in Sauget, Illinois (Figures 1.1 and 1.2). Solutia Inc. signed the Administrative Order on Consent, Docket No. R8H-5-00-003, on May 26, 2000. Sections VI.1a, 1b, 2, 3 and 5, respectively, required Solutia to submit a Description of Current Conditions Report, investigate the nature and extent of any releases at or from the W.G. Krummrich facility, stabilize groundwater migration and show that any discharge of groundwater to surface water is either insignificant or currently acceptable, control completed pathway human exposures to contamination and propose final corrective measures for the site.

To fulfill the requirements of the AOC Solutia submitted a Description of Current Conditions Report, performed site investigations for air, soil, DNAPL and groundwater, completed Environmental Indicator Determinations for Migration of Contaminated Groundwater under Control (CA750) and Current Human Exposure Under Control (CA725) and submitted a Final Corrective Measures Study as summarized in the following table:

Summary of Work Performed to Fulfill the Requirements of the W.G. Krummrich RCRA AOC (Docket No. R8H-5-00-003)

• Description of Current Conditions Report	August 1, 2000
• Sediment, Surface Water and Fish Tissue Sampling	October and November 2000
• Ecological Risk Assessment	June 1, 2001
• CA750 Migration of Contaminated Groundwater Under Control Environmental Indicator Determination	May 26, 2004
• CA725 Current Human Exposure Under Control Environmental Indicator Determination	May 26, 2004
• Air, Soil, DNAPL and Groundwater Investigation	2003 and 2004
• Corrective Measures Study	August 27, 2004

In addition to these actions, Solutia implemented or planned a number of removal and remedial actions at Sauget Area 1, Sauget Area 2 and the W.G. Krummrich Facility prior to and after the May 26, 2000 RCRA AOC. A time line of the various removal actions and remedial actions and estimated expenditures for each action are given below:

Time Line of Sauget Area 1, Sauget Area 2 and W.G. Krummrich Removal/Remedial Actions and Estimated Expenditures

Sauget Area 1	2001	Dead Creek Culvert Replacement Removal Action	$750,000
	2002	Dead Creek Time Critical Sediment Removal Action	12,300,000
	2004	Dead Creek Segment B, D and F Soil Removal Action Plan	
Sauget Area 2	1979	Site R Capping	
	1985	Site R Riverbank Stabilization	750,000
	2003/4	Groundwater Migration Control System	25,400,000
W.G. Krummrich	1987	Route 3 Drum Site Impermeable Cap	
	2000	Sewer System Improvements	17,100,000
	2001	Chlorobenzene Process Area Spill	
	2003	Plant Process Area Permeable Covers	310,000
		Estimated Total Expenditure	$56,610,000

Solutia prepared a Plume Stability Monitoring Plan as part of the CA-750 RCRA Environmental Indicator

program, and submitted it to USEPA on April 19, 2004. On December 3, 2004, USEPA issued comments to this plan. In addition, some of USEPA's comments on the CMS Report (November 18, 2004 comment letter) addressed groundwater quality. In the monitoring plan, submitted with the Response to Comments for the CMS on February 9, 2005 (CMS RTC), Solutia addressed both sets of comments, as they pertain to groundwater quality monitoring and plume stability.

On May 4, 2005, USEPA issued final comments to the CMS RTC; some of which pertained to the groundwater monitoring plan. Specifically, Comment 14 – Section 7.2, requested "a stand alone work plan for the groundwater monitoring program that adequately addresses comments on Section 6 in this Enclosure and comments previously provided to Solutia in a letter dated December 3, 2004." In response to that request, Solutia is submitting this stand alone work plan for the plume stability monitoring program. USEPA's August 16, 2005 comments are also addressed in this work plan.

This Plume Stability Monitoring Plan, developed to comply with the CA-750, includes the following sections:

Section 1.0	**Introduction**
Section 2.0	**Monitoring Well Installation and Development**
Section 3.0	**Water Level Gauging and Groundwater Sampling**
Section 4.0	**Groundwater Data Management, Evaluation and Reporting**
Section 5.0	**Groundwater Monitoring Schedule**
Section 6.0	**References**

Monitoring wells will be installed at 17 locations and sampled as part of the Plume Stability Monitoring Well network. The monitoring well locations include source area wells and downgradient wells within and along the groundwater contamination plume boundaries as defined from groundwater monitoring conducted to support the CA-750, particle track modeling conducted for the CMS, and modeling of site-wide groundwater quality data collected from 2002 to 2004.

This Plume Stability Monitoring Plan describes the procedures for: well installation; groundwater level measurements; collection of groundwater samples, sample analysis, and information on reporting. In addition, the plan provides objectives, organization, functional activities, and specific Quality Assurance (QA) and Quality Control (QC) activities for sampling, sample handling and storage, chain of custody, and laboratory and field analysis efforts associated with sampling of environmental media in accordance with the USEPA Region 5 Model Quality Assurance Project Plan (QAPP). As such, this Work Plan includes elements associated with Field Sampling Plans and QAPPs.

2.0 MONITORING WELLS

2.1 Monitoring Well Location

Plume stability monitoring well locations were determined using on CA-750 groundwater quality data, particle track modeling conducted for the CMS, and modeling of site-wide groundwater quality data collected from 2002-2004. This work defined the flowpaths of groundwater from the facility downgradient to the Mississippi River including definition of groundwater flowpaths that would be captured by the Sauget Area 2 Groundwater Migration Control System (GMCS), as well as flowpaths that would not be captured by the GMCS. The plume stability network of 17 wells is comprehensive in that it includes source area wells and downgradient wells within and along the plume boundaries selected to monitor plume boundary conditions and plume stability. The table below describes the plume stability monitoring well location criteria and well locations are shown on Figure 2.1.

Plume Stability Monitoring Well Location Criteria

Monitoring Area	Monitoring Wells	Plume Stability Monitoring Well Location Criteria
Source Areas	2, 3, 4 and 5	Located in Source Areas in Plant Process Area • PSMW 2 - Former PCB Manufacturing Area • PSMW 3 - Former Chlorobenzene Process Area • PSMW 4 - North Tank Farm Area • PSMW 5 - Former Chlorobenzene Storage Area
Northern Plume Boundary	1, 6, 10 and 14	Provide Data along Northern Plume Boundary
Chlorobenzene Process Area Groundwater Migration Pathway	7, 11 and 15	Groundwater Migration Pathway Downgradient of Former Chlorobenzene Process Area (Includes PSMW-3)
Chlorobenzene Storage Area Groundwater Migration Pathway	8, 12 and 16	Groundwater Migration Pathway Downgradient of Former Chlorobenzene Storage Area (Includes PSMW-5)
Southern Plume Boundary	9, 13 and 17	Provide Data along Southern Plume Boundary

2.2 Monitoring Well Depth

Historical groundwater quality data from 1983 to 2000 and groundwater quality data collected between 2002 and 2004 for the CA-750 Environmental Indicator Determination were used to selected screen depths for the plume stability monitoring wells. In general, these data indicate that the highest groundwater concentrations are typically found in the Shallow Hydrogeologic Unit (SHU) and Middle Hydrogeologic Unit (MHU) while the greatest areal extent of impacted groundwater occurs in the Deep Hydrogeologic Unit (DHU). This distribution of constituents in groundwater appears to be due to two primary causes: 1) higher concentrations of DNAPL on soils in the SHU and MHU than in the DHU and 2) higher permeability in the DHU than the SHU and MHU.

2.2.1 Groundwater Concentrations in the SHU and MHU

Only three existing monitoring well clusters that sample the SHU, MHU and DHU are located in the plant process area: GM-9 A, B and C; GM10 A, B and C and GM-12 A, B and C. Historical groundwater quality data from these monitoring wells are summarized below and the highest detected concentrations are highlighted:

Maximum Detected Concentrations in Existing Monitoring Wells GM-9 A, B and C, GM-10 A, B and C and GM-12 A, B and C

	1983 to 1986		1986 - 1994		2000	
Monitoring Well (ft bgs)	Total VOCs (ug/l)	Total SVOCS (ug/l)	Total VOCs (ug/l)	Total SVOCs (ug/l)	Total VOCs (ug/l)	Total SVOCs (ug/l)
GM-9 A (13 - 28)	1,185	225	3,380	NA	11	54
B (55 - 75)	2,159	127	4,446	NA	133	80
C (88-108)	291	140	2,037	NA	51	28
GM-10 A (13 - 28)	55	8	5,030	3,086	ND	8
B (54 - 74)	1,857	1,499	3,140	ND	2,480	1,970
C (94 - 109)	107	31	752	36	2,960	469
GM-12 A (18.5 - 33.5)	5,288	1,107	4,830	3,086	2,056	35,585
B (69 - 89)	2,573	207	9,400	ND	7,200	255
C (94 - 114.5)	232	672	3,960	36	340	32

As can be seen from these data, the highest detected Total VOC and Total SVOC concentrations are found in the SHU and MHU with the exception of GM-10C where Total VOCs where highest in the DHU in 2000.

Data from the DNAPL investigation performed for the W.G. Krummrich Corrective Measures Study indicates that DNAPL concentrations in soil are typically higher in the SHU and MHU than in the DHU:

Total VOC and Total SVOC Concentrations in Soils Within and Adjacent to the Chlorobenzene Process Area

	DNAPL Boring K - 3		DNAPL Boring K - 4		DNAPL Boring K - 5	
Sample Depth (ft bgs)	Total VOCs (mg/kg)	Total SVOCS (mg/kg)	Total VOCs (mg/kg)	Total SVOCs (mg/kg)	Total VOCs (mg/kg)	Total SVOCs (mg/kg)
0 - 10	181	313	1,601	15,020	253	260
10 - 20	2,423	4,371	131	5,375	16,800	31
20 - 30	208	1,303	464	153	9,600	213
30 - 40	390	144	28	47	100	3.7
Typical Bottom of Shallow Hydrogeologic Unit						
40 - 50	588	2,005	32	140	1,349	6.4
50 - 60	58	60	15	9	4.2	1.6
60 - 70	40	118	8	3	<1	1.2
70 - 80	97	576	134	68	<1	1.2
Typical Bottom of Middle Hydrogeologic Unit						
80 - 90	<1	26	<1	28	<1	ND
90 - 100	<1	<1	1.9	16	<1	<1
100 - 110	<1	1.3	<1	<1	NS	NS
110 - 120	NS	NS	11	253	10	<1
Typical Bottom of Deep Hydrogeologic Unit						

Notes:
1) Typical depths below ground surface: SHU 0 to 40
MHU 40 to 80
DHU 80 to 120

Total VOC and Total SVOC concentrations range from 28 to 16,800 mg/kg and 3.7 to 15,020 mg/kg, respectively, in the SHU; <1 to 1,349 mg/kg and 1.2 to 2,005 mg/kg, respectively, in the MHU and <1 to 10 mg/kg and <1 to 253, respectively, in the DHU. Based on these data, DNAPL concentrations in soil decrease with depth, with the highest concentrations in the SHU and MHU and lowest in the DHU.

This vertical distribution of constituent concentrations in soil is the same as observed in groundwater - high concentrations shallow, low concentrations deep. Higher concentrations of site constituents in groundwater in the SHU and MHU than in the DHU appear to be the result of DNAPL dissolution. Constituents that are typically detected in site groundwater include benzene, chlorobenzene, dichlorobenzene, chloroaniline, phenol, chlorophenol, dichlorophenol and napthalene. Maximum detected concentrations of these constituents in soil samples collected from DNAPL borings are given below along with their solubility limits in water:

Maximum Detected Soil Concentrations in Chlorobenzene Process Area DNAPL Borings K-3, K-4 and K-5

Constituent	Soil Concentration (mg/kg)	Solubility Limit (mg/l)
Dichlorobenzene	4,200	156
Chlorobenzene	2,300	472
Chloroaniline	97	3,900
Benzene	23	1,791
Dichlorophenol	12	4,600
Phenol	0.8	87,000
Napthalene	0.5	30
Chlorophenol	0.4	28,000

The primary constituents detected in site groundwater are present in site soils, and all of these constituents are soluble in water to a greater or lesser degree. Consequently, it seems reasonable to conclude that DNAPL dissolution is a major, if not the primary source, of constituent concentrations observed in site groundwater. Since soil concentrations of these constituents are highest in the SHU and MHU, it stands to reason that groundwater concentrations of these constituents would be highest in the SHU and DHU, which is indeed the case.

2.2.2 Groundwater Concentrations in the DHU

Isoconcentration maps for BTEX, benzene, chlorobenzene and dichlorobenzene; phenol, chlorophenol and dichlorophenol; and chloroaniline were prepared for this work plan using CMS groundwater quality data and Environmental Visualization System software (EVS, Version 7.92). Groundwater quality data is presented in Appendix A and the groundwater isoconcentration maps are presented in Appendix B. These isoconcentration maps also include groundwater data from the DNAPL piezometers and ground surface and bedrock surface elevations.

Comparison of the areal distribution of benzene, chlorobenzene, chloroanilines, chlorophenols, dichlorobenzenes, phenols and BTEX in the SHU, MHU and DHU (Figures 2.2 through 2.8) demonstrates that the greatest areal extent of these constituents occurs in the DHU. Preferential flow of site-related constituents through the DHU is most likely the result of higher permeability in this hydrogeologic unit than in the SHU and DHU. Average hydraulic properties of the unconsolidated aquifer, based on 10 aquifer tests and 100 specific capacity tests conducted on industrial, municipal, irrigation and relief wells in the American Bottoms over a span of 30 years, are:

Average Hydraulic Properties of the American Bottoms Aquifer

Hydrogeologic Unit	Transmissivity (gpd/ft)	Hydraulic Conductivity (gpd/ft^2)	Storage Coefficient
• **Shallow Hydrogeologic Unit**	**141.5**	**9.5**	**Not Available**
• **Middle Hydrogeologic Unit**	**165,000**	**3,300**	**0.04**
• **Deep Hydrogeologic Unit**	**211,000**	**2,600**	**0.002 to 0.100**

In metric units, the SHU, MHU and DHU have hydraulic conductivities of 4×10^{-4}, 1.6×10^{-1} and 1.2×10^{-1} cm/sec, respectively.

Slug tests performed in Sauget Area 1 indicate that the average permeability of the SHU, MHU and DHU are 8.9×10^{-3}, 3.5×10^{-2} and 5.4×10^{-2} cm/sec, respectively. Based on these data, the permeability of the MHU is a factor of 4.3 higher than the SHU and permeability of the DHU is a factor of 1.5 higher than the MHU. Slug tests performed for the W.G. Krummrich CMS indicates that permeability in the MHU is a factor of 2.8 higher in the MHU than the SHU (2.5×10^{-2} versus 9.0×10^{-3} cm/sec) and that permeability in the DHU is a factor of 1.3 higher than in the MHU (3.3×10^{-2} versus 2.5×10^{-2} cm/sec).

These hydraulic conductivity contrasts appear to control the areal extent of migration. Figure 2.2 shows the areal extent of benzene in the SHU, MHU and DHU. Benzene in the SHU is confined to the W.G. Krummrich facility while it extends approximately 600 feet downgradient of Lot F in the MHU and extends to the Mississippi River in the DHU. The areal extent of benzene in the SHU, MHU and DHU is approximately 172 acres, 177 acres and 415 acres, respectively, based on the areal extent of the 1 ug/l isocon. Chlorobenzenes extend to the downgradient limit of Lot F in the SHU, about 1000 feet downgradient of Lot F in the MHU and reach the Mississippi River in the DHU (Figure 2.3). The areal extent of chlorobenzene in the SHU, MHU and DHU is approximately 282 acres, 508 acres and 914 acres, respectively. Chloroanilines show a slightly different pattern (Figure 2.4). They are not present in the SHU on site but extend to the downgradient limit of Lot F in the MHU. The farthest extent of downgradient migration is in the DHU where chloroanilines extend 1800 feet downgradient of Lot F. The areal extent of chloroanilines in the SHU, MHU and DHU is approximately 12 acres, 78 acres and 292 acres, respectively,

Chlorophenols are present in the SHU at the downgradient limit of the plant production area (Figure 2-5) and extend 700 feet downgradient of Lot F in the MHU and 1900 feet downgradient in the DHU. The areal extent of chlorophenols in the SHU, MHU and DHU is approximately 115 acres, 92 acres and 517 acres, respectively. Dichlorobenzenes in the SHU are located in two areas of the plant: 1) at the southern end of the Former Chlorobenzene Process Area and 2) the Former Chlorobenzene Storage Area (Figure 2-6). They do not extend past Lot F in the MHU and extend 1900 feet downgradient of Lot F in the DHU. The areal extent of dichlorobenzenes in the SHU, MHU and DHU is approximately 23 acres, 115 acres and 437 acres, respectively,

Phenol is present in the SHU at the downgradient limit of the plant process area, does not extend past the downgradient limit of Lot F in the MHU and extends 800 feet downgradient of Lot F in the DHU (Figure 2-7). Phenol is also present in the MHU and DHU at the upgradient end of the plant process area and the DHU north of the Former Chlorobenzene Process Area. The areal extent of phenol in the SHU, MHU and DHU is approximately 69 acres, 92 acres and 115 acres, respectively.

The areal distribution of BTEX in the SHU, MHU and DHU is similar to benzene (Figure 2-8). The areal extent of BTEX in the SHU, MHU and DHU is approximately 126 acres, 218 acres and 471 acres, respectively,

Based on this information, site-related constituents are migrating preferentially through the DHU, resulting in the farthest downgradient extent of migration and the largest area of impacted groundwater of any of the three hydrogeologic units found at the W.G. Krummrich facility.

2.3 Monitoring Well Screen Depth

Chlorobenzene (MCB) and Dichlorobenzenes (DCB) have the largest areal extent of any of the site-related constituents present in groundwater (Figures 2-3 and 2-6). Because their distribution includes the area occupied by other constituents and they are the constituents that migrated furthest from the facility and are closest to the Mississippi River, MCB and DCB were used to select plume stability monitoring well screen depths.

Selected screen depths for the W.G. Krummrich Plume Stability Monitoring Plan are given in Tables 2.1 to 2.15. These screen depths, which are based on the highest detected groundwater concentrations in the nearest vertical profile to the plume stability monitoring well location (Figure 2.9), are summarized below:

Summary of Selected Plume Stability Monitoring Well Screen Depths (Feet below Ground Surface)

Well	Screen Depth	Well	Screen Depth	Well	Screen Depth	Well	Screen Depth
MW-1	43	MW-6	110	MW-11	104	MW-16	120
MW-2	72	MW-7	119	MW-12	100	MW-17	127
MW-3	72	MW-8	73	MW-13	114		
MW-4	97.5	MW-9	104	MW-14	112		
MW-5	22.5	MW-10	104	MW-15	112		

Note: Depth below ground surface to midpoint of 5 ft. long screen

Plume stability monitoring wells PSMW-1, 2, 3, 4 and 5 are located in source areas and, as a consequence, selected screen depths are in the lower portion of the SHU and the MHU. GM-17 A, B and C is the closest groundwater vertical profile to PSMW-5. The highest detected Total VOC and Total SVOC concentrations at GM-17 A, B and C occur in GM-17 A, which is screened 18 to 38 ft. below ground surface. Based on this information, the screen in PSMW-5 should be installed between 18 and 38 ft. below ground surface. Historical groundwater data (2001) from GM-32, 33 and 34, which are located in the same area as GM-17 A, B and C and PSMW-5, indicate that Total VOC concentrations in wells GM-32, 33 and 34 range from 326,000 to 1,600,000 ug/l. GM-32 is screened from 9 to 24 ft. below ground surface while GM-33 and 34 are screened from 5 to 25 ft. below ground surface. Because the historical groundwater data from GM-32, 33 and 34 indicate that the highest Total VOC concentrations occur from 5 to 25 ft. below ground surface in the area of PSMW-5, this monitoring well will be screened from 5 to 25 ft. below ground surface instead of 18 to 38 ft. below ground surface. To ensure that groundwater quality samples can be collected from PSMW-5 during dry weather conditions, the well screen will be installed from 20 to 25 ft. below ground surface. Depth to groundwater is typically no more than 15 ft. below ground surface but can reach 20 ft. below ground surface in dry weather conditions.

The MHU will also be screened at PSMW-8 because VOC and SVOC concentrations are highest in this hydrogeologic unit, probably as a result of past spills and leaks from the Former Chlorobenzene Storage Area.

Screens will be installed in the DHU at PSMW-6, 7, 9, 10, 11, 12, 13, 14, 15, 16 and 17. These monitoring wells are located downgradient of the Plant Process Area where the highest groundwater concentrations are found in the deeper portions of the alluvial aquifer.

Screen depths generally get deeper with increasing distance from the W.G. Krummrich facility downgradient boundary. Screen depths at PSMW-6, 7, 8 and 9, located at the downgradient boundary of the plant, average 101.5 feet and range from 73 to 119 ft bgs. Monitoring wells PSMW-10, 11, 12 and 13, which are located approximately half way between the plant boundary and the Mississippi River, have an average screen depth of 104.5 ft bgs and range from 100 to 114 ft bgs. At the Mississippi River, PSMW-14, 15, 16 and 17 have an average depth of 117.75 ft bgs and range from 112 to 127 ft bgs.

This screen depth pattern reflects high concentrations in shallow groundwater in the source areas and high concentrations in deep groundwater in the discharge area as groundwater migrates from the plant and discharges to the Mississippi River.

At location PSMW-2, in the Former PCB Manufacturing Area, MCB and DCB vertical distribution (Tables 2.2 and 2.3) indicates that the well screen midpoint should be 72 to 73 ft. below ground surface. However, there is insufficient data for PCBs in groundwater to identify the proper screen interval. Solutia plans to address this data gap by conducting groundwater profiling in this area. A separate work plan will be developed and submitted to USEPA for this work.

If property access is denied for these proposed locations, monitoring wells will be installed on the closest available property where if the location would still meet the objectives of the groundwater monitoring program.

2.4 Monitoring Well Installation

URS Corporation (URS) will perform the field activities in accordance with this plan, Standard Operation Procedures (SOPs), and an approved Health and Safety Plan (HASP). URS will coordinate with Solutia personnel to obtain the appropriate permits and clearance to perform the subsurface activities.

The monitoring wells will be installed using rotasonic drilling techniques. The borings will be advanced using an approximately 4-inch diameter by 10-foot long core barrel and a 6-inch diameter override casing. Soil cores will be continuously collected from the core barrel through the length of the boring. The monitoring wells will be installed through the six-inch override casing. Plume stability monitoring wells PSMW-14M, 15M and 16M will be installed in the borehole as wells PSMW-14D, 15D and 16D. Drill cuttings will be containerized and staged per Solutia procedures.

The subsurface stratigraphy will be logged during drilling operations by a qualified field scientist in accordance with the Unified Soil Classification System (USCS) protocols. The field scientist will note soil attributes such as color, particle size, consistency, moisture content, structure, plasticity, odor (if obvious) and organic content (if visible). Soil samples from each boring will be visually evaluated for evidence of impact and screened in the field using a Photoionization Detector (PID). Information pertaining to the subsurface soil and drilling conditions will be recorded in the field on a standard field boring log form in accordance with the SOP-8 (Appendix C). Representative scaled, color digital photographs will be taken of each soil core to provide a record of materials present at this site.

Ground surface elevation will be confirmed at each vertical groundwater profile location and determined for each plume stability monitoring well location prior to well installation. Ground surface elevation at each monitoring well location will then be compared to ground surface elevation at the vertical

groundwater profile locations used to select plume stability monitoring well screen depths. These data will then be used to determine the depth below ground surface of the midpoint of each 5 ft.-long plume stability monitoring well screen. Vertical groundwater profile elevations are listed below:

Vertical Groundwater Profile Ground Surface Elevations, Ft. AMSL

Monitoring Well	Vertical Profile	Elevation	Monitoring Well	Vertical Profile	Elevation
PSMW - 1	TRA1-GPB	413	PSMW - 10	AA-P-1	408
PSMW - 2	TRA2-GPC and D	411	PSMW - 11	AA-P-1	408
PSMW - 3	TRA2-GPD	411	PSMW - 12	AA-GWM-S1	422
PSMW - 4	GM-4A, B and C	404.5		AA-GWM-S2	410.5
PSMW - 5	GM-17A, B and C	410	PSMW - 13	AA-S-1B	411
PSMW - 6	UAA-1	406	PSMW - 14	AA-P-3	410.5
PSMW - 7	TRA4-GPB	416	PSMW - 15	AA-P-3	410.5
PSMW - 8	TRA4-GPC	413	PSMW - 16	AA-GWM-S1	422
PSMW - 9	UAA-3	405	PSMW - 17	AA-Q-1	420

Notes: Elevations in feet above mean sea level

Well screen depths (midpoint of 5 ft. long screens) and screened intervals are provided in the table below.

Monitoring Well No.	Monitoring Area	Estimated Depth Below Ground Surface	Monitoring Well Screen Midpoint Elevation
PSMW - 1	Northern Plume Boundary	43	370
PSMW - 2	Source Area - PCB Mfg. Area	72	339
PSMW - 3	Source Area - CPA	72	339
PSMW - 4	Source Area -North Tank Farm	97.5	307
PSMW - 5	Source Area - CSA	22.5	387.5
PSMW - 6	Northern Plume Boundary	110	296
PSMW - 7	CPA Migration Pathway	119	297
PSMW - 8	CSA Migration Pathway	73	340
PSMW - 9	Southern Plume Boundary	104	301
PSMW - 10	Northern Plume Boundary	104	304
PSMW - 11	CPA Migration Pathway	104	304
PSMW - 12	CSA Migration Pathway	100	306
PSMW - 13	Southern Plume Boundary	114	297
PSMW - 14 M	Northern Plume Boundary	45.5	365
PSMW - 14 D	Northern Plume Boundary	112	298.5
PSMW - 15 M	CPA Migration Pathway	45.5	365
PSMW - 15 D	CPA Migration Pathway	112	298.5
PSMW - 16 M	CSA Migration Pathway	60	365
PSMW - 16 D	CSA Migration Pathway	120	302
PSMW - 17	Southern Plume Boundary	127	293

Note: PSMW-12 screen midpoint elevation based on average of grade elevation and selected screen depth at AA-GWM-S1 (422 ft amsl and 120 ft bgs, respectively) and grade elevation and selected screen depth at AA-GWM-S2 (410.5 ft amsl and 100 ft bgs, respectively).

At the completion of the boring, the monitoring well will be constructed in accordance with state of Illinois guidelines by a permitted Illinois well driller. Monitoring wells will be constructed of two-inch diameter

Type 304 stainless steel casings, with a five foot section of 0.010-in. stainless steel well screen (continuous wrap). A sand filter pack consisting of silica sand will be installed through the casing from the base of the well and extended to approximately 2-foot above the top of the well screen. During placement of the sand pack, the height will be checked periodically to ensure that the volume placed within the annulus correlates to the calculated volume required to fill the annular space. A bentonite seal with a minimum thickness of 2-foot will be installed directly above the sand pack. The remaining annular space will be filled with a cement/bentonite or high solids grout. The surface completion of the monitoring wells will include placement of a concrete pad, installation of locking caps and stickup or flush mount well covers, and placement of bumper posts, as necessary.

Monitoring well construction information will be documented on the boring logs, monitoring well construction diagrams. The locations will be surveyed to obtain horizontal and vertical coordinates. SOPs for drilling and well installation are included in Appendix C.

2.5 Monitoring Well Development

The monitoring wells will be developed to remove the fines from the well and sand pack. This will be performed using a conventional groundwater pump, air-lift system, or equivalent methods suitable for well development (SOP-5). Each monitoring well will be developed until a minimum of five well volumes have been removed and pH, specific conductance, and temperature readings stabilize within 10% over a minimum of two successive readings. In addition the turbidity of the development water will be recorded to ensure that fines have been removed. The field measurements will be measured and recorded on monitoring well sampling sheets during development.

2.6 Decontamination and Investigation Derived Waste

Field personnel and equipment will undergo decontamination procedures to ensure the health and safety of those present, to maintain sample integrity, and to minimize the movement of contamination between the work area and off-site locations. Equipment used on-site will be decontaminated prior to beginning work, between sampling locations and/or uses, and prior to demobilizing from the site. Non-disposable purging and sampling equipment will be decontaminated between each sample acquisition by washing with an Alconox® or equivalent detergent wash, a potable water rinse, and a distilled water rinse. Personnel and small equipment decontamination will be performed at the sample locations. Disposable sampling equipment, such as gloves will be collected and bagged on a daily basis and managed in accordance with Solutia procedures. Soil cuttings will be containerized and staged per Solutia procedures. Development water will be containerized and handled per Solutia procedures. Refer to SOP-6 in Appendix C of this plan for decontamination procedures.

3.0 GROUNDWATER MONITORING

3.1 Groundwater Monitoring Frequency

The monitoring well network will be sampled on a quarterly basis for a minimum of two years to develop a baseline for assessing plume stability. The baseline will be used to make plume stability determinations for future monitoring.

3.2 Groundwater Level Gauging

Prior to each sampling round, groundwater level and non-aqueous phase liquid (NAPL) (potential) measurements will be obtained from the available existing piezometers clusters installed for the Sauget Area 1 EE/CA and RI/FS, the Sauget Area 2 RI/FS and the WGK CA-750 Environmental Indicator projects. The locations of these piezometers are shown on Figure 3.1. The piezometers have been assigned unique-identification numbers for the purposes of this plan. Table 3.1 summarizes construction information for these piezometers.

Construction information for piezometer clusters GWE-11, 12, 13, 16, 17, 18, 19, 20 and 21 are not included in Table 3.1 because well construction records are not available for these piezometers. As part of the first groundwater sampling round, these piezometers will be probed in an attempt to determine their depths and screen zones. This information will be incorporated into the groundwater level piezometer and well construction summary table. However, it is known that three one-inch diameter piezometers were installed at each cluster location with one piezometer screened at the top of the SHU (20 ft bgs), a second screened at the top of the MHU (40 ft bgs) and a third screened at the top of the DHU (60 ft bgs). Five-foot long screens were installed in each piezometer. Top of casing elevations are given below:

Top of Casing Elevations for Piezometer Clusters GWE-11, 12, 13, 16, 17, 18, 19, 20 and 21

Piezometer Cluster	Shallow	Middle	Deep
GWE-11	416.69	416.70	416.65
GWE-12	414.83	414.97	414.90
GWE-13	415.92	415.94	415.97
GWE-16	410.87	410.57	410.90
GWE-17	407.52	407.44	407.60
GWE-18	409.26	409.10	409.48
GWE-19	411.81	411.68	411.85
GWE-20	410.11	409.81	410.15
GWE-21	412.01	412.08	412.16

Groundwater levels will also be measured in the groundwater monitoring wells installed as part of this work. These data will be used to develop groundwater elevation contour maps for each hydrostratigraphic zone.

3.3 Groundwater Sampling Parameters

Groundwater sampled during the first sampling round will be analyzed for RCRA hazardous constituents, specifically 40 CFR Appendix IX VOCs (Method 8260B), SVOCs (Method 8270C), PCBs (Method 680), Pesticides (Method 8081A), Herbicides (Method 8151A), and Metals (Method 6010B). Past groundwater sampling indicates that the primary constituents migrating from source areas to or toward the Mississippi River are MCB (Method 8260B) and DCB (Method 8270C); therefore for subsequent events, monitoring parameters will focus on MCB and DCB, the constituents most indicative of plume conditions and stability, based on historical VOC and SVOC analysis, and other parameters to assess the effectiveness of natural attenuation. Based on the results of the initial sampling, a focused analytical program will be proposed for future events. Severn-Trent Laboratories (STL) Savannah, Georgia will provide analytical services for this monitoring plan. Chemical analysis will be conducted in accordance with the laboratory SOPs located in Appendix D. The proposed parameters are summarized below:

Location	Monitoring Well No.	Laboratory Analyses
North Tank Farm, Northern and Southern Plume Boundaries	1, 4, 6, 10, 14, 9, 13, 17	VOCs (Method 8260B) SVOCs (Method 8270C) PCBs (Method 680) Pesticides (Method 8081A) Herbicides (Method 8151A) and/or Metals (Method 6010)
Former Chlorobenzene Process Area Groundwater Migration Pathway	3, 7, 11, 15	VOCs (Method 8260B) SVOCs (Method 8270C) PCBs (Method 680) Pesticides (Method 8081A) Herbicides (Method 8151A) and/or Metals (Method 6010) MNA parameters - Alkalinity, carbon dioxide, chloride, ferrous iron, methane, nitrate, sulfate, total organic carbon
Former Chlorobenzene Storage Area Groundwater Migration Pathway	5, 8, 12, 16	VOCs (Method 8260B) SVOCs (Method 8270C) PCBs (Method 680) Pesticides (Method 8081A) Herbicides (Method 8151A) and/or Metals (Method 6010) MNA parameters - Alkalinity, carbon dioxide, chloride, ferrous iron, methane, nitrate, sulfate, total organic carbon
Former PCB Manufacturing Area	2	VOCs (Method 8260B) SVOCs (Method 8270C) PCBs (Method 680) Pesticides (Method 8081A) Herbicides (Method 8151A) and/or Metals (Method 6010)

In Solutia's May 27, 2005 responses to USEPA's May 4, 2005 comments, Solutia agreed to analyze samples for MCB and DCB aerobic biodegradation products, if possible using Methods 8260B and

8270C. Based on conversations with STL Savannah, the biodegradation products listed in the May 27, 2005 letter cannot be identified using standard test methodologies.

Bacterial plate counts will be performed annually to determine the number of colony forming units present in the most affected well.

3.4 Groundwater Sampling Procedures

Groundwater level measurements and samples will be obtained from the seventeen monitoring wells illustrated on Figure 2.1. The twenty wells will be sampled on a quarterly basis for two years and then the sampling program will be reevaluated. Samples will be analyzed as described in Section 3.3 above.

Groundwater samples will be collected using low-flow methodologies including a flow-through cell. General procedures for low-flow sample collection are described below. Additional details are included SOP-7 and SOP-8 located in Appendix C. The groundwater sampling will proceed from the least impacted wells to the most impacted. Equipment used for sampling that could contact groundwater will be properly decontaminated before each use. Field instruments will be calibrated prior to use in accordance with the manufacturer's specifications.

Before well purging begins, the well will be inspected for security, damage, and evidence of tampering. If damage or tampering is evident, the project manager will be contacted for guidance. Clean plastic sheeting will be placed around the well and ambient volatile organic compound (VOC) background levels in the immediate vicinity of the well will be measured (i.e., using a PID or a flame ionization detector (FID)). Once the well cap is removed, VOCs will be measured at the rim of the well and the readings recorded in the logbook or on the groundwater sampling form.

Immediately prior to sampling, groundwater elevations (and the presence of any NAPL) will be measured to the nearest 1/100 ft using an electronic interface probe and documented. If NAPL is present, efforts will be made to collect water above or below the NAPL. The depth to the bottom of each well will be measured immediately after sampling, to minimize disturbing the water column. The monitoring well information for the wells to be sampled, including screen intervals are summarized below:

Monitoring Well No.	Monitoring Area	Estimated Depth Below Ground Surface	Monitoring Well Screen Midpoint Elevation
PSMW - 1	Northern Plume Boundary	43	370
PSMW - 2	Source Area - PCB Mfg. Area	To Be Determined	To Be Determined
PSMW - 3	Source Area - CPA	72	339
PSMW - 4	Source Area -North Tank Farm	97.5	309.5
PSMW - 5	Source Area - CSA	22.5	386.5
PSMW - 6	Northern Plume Boundary	110	296

Monitoring Well No.	Monitoring Area	Estimated Depth Below Ground Surface	Monitoring Well Screen Midpoint Elevation
PSMW - 7	CPA Migration Pathway	119	288
PSMW - 8	CSA Migration Pathway	73	340
PSMW - 9	Southern Plume Boundary	104	301
PSMW - 10	Northern Plume Boundary	104	306
PSMW - 11	CPA Migration Pathway	104	304
PSMW - 12	CSA Migration Pathway	100	316
PSMW - 13	Southern Plume Boundary	114	297
PSMW - 14 M	Northern Plume Boundary	45.5	365
PSMW - 14 D	Northern Plume Boundary	112	298.5
PSMW - 15 M	CPA Migration Pathway	45.5	365
PSMW - 15 D	CPA Migration Pathway	112	298.5
PSMW - 16 M	CSA Migration Pathway	60	365
PSMW - 16 D	CSA Migration Pathway	120	305
PSMW - 17	Southern Plume Boundary	127	293

The monitoring wells will be purged using a conventional groundwater pump, suitable for low flow applications (i.e., bladder pump [or equivalent]). Prior to purging the pump will slowly be lowered to a depth in the well as described in the SOP. When purging first begins, the pump flow rate will be started at approximately 100mL/min or the lowest flow rate possible. Water level measurements and flow rate measurements will be taken every 2 minutes until they indicate that significant drawdown within the well is not occurring. Measurements will be scaled back to every five minutes when drawdown reaches equilibrium. Ideally, drawdown will be limited to 25% of the distance between the top of the screen and the pump intake. If significant drawdown occurs, the well will be pumped dry. After being pumped dry, the well will be periodically gauged until the water level has recharged to approximately 90% of the original, static level prior to sampling. If in 24 hours the well has not reached 90% static recovery, the well will be sampled.

Each monitoring well will be purged until pH, specific conductance, dissolved oxygen and oxygen reduction potential stabilize over a minimum of three successive flow-through cell volumes. In addition temperature and turbidity will be measured but not used as sampling criteria. The field parameters will be measured and recorded on monitoring well sampling sheets during purging. The allowable ranges for the criteria used to determine stabilization is provided below:

- **pH** **+/- 0.2 units**
- **Conductivity** **+/- 3%**
- **DO** **+/- 10% or +/-0.2 mg/L, whichever is greatest**
- **ORP** **+/- 20 mV**

After the relevant parameters have stabilized, the flow-through cell will be bypassed for sampling. Groundwater samples will be collected at a flow rate no greater than 0.5 L/min (to minimize aeration)

using the same pump used for purging. Personnel conducting the groundwater sampling will wear clean disposable protective gloves. Sample containers will be filled in the order below:

- **VOC vials should be filled completely so that the water forms a convex meniscus then capped so that no air space exists in the vial. Turn the vial over and tap it to check for bubbles. If air bubbles are observed in the sample vial, remove the lid and attempt to fill the vial two more times, (being careful not to dump out any groundwater currently in the vial). If air bubbles are present twice more, discard the sample vial and repeat the procedure with a new vial. If, after three attempts, air bubbles are still in the vial, make a note of this and place the vial in the cooler.**
- **Gas sensitive parameters (e.g., ferrous iron, methane, alkalinity)**
- **Semivolatile organic compounds**
- **Pesticides**
- **Polychlorinated biphenyls**
- **Herbicides**
- **Metals (unfiltered)**

To verify field and laboratory procedures, quality assurance/quality control (QA/QC) samples consisting of duplicate samples, matrix spike/matrix spike duplicate (MS/MSD) samples, equipment blanks and trip blanks will be collected and submitted to the laboratory. QA/QC samples will be collected at a frequency of 10% for duplicates and blanks and 5% for MS/MSDs. One trip blank will accompany each cooler containing VOC analysis samples.

Equipment test checks are to be used to ensure QA/QC for the equipment used in field work. In order to reduce the potential for exposure to hazardous materials and limit the possibility of cross contamination of samples, field personnel and equipment will comply with decontamination and IDW procedures as described in Section 2.6 of this plan.

For proper identification in the field and proper tracking by the analytical laboratory, investigative and QC samples will be labeled in a clear and consistent fashion. Sample labels will be waterproof, or sample containers will be sealed in plastic bags. Field personnel will maintain a sampling log sheet containing information sufficient to allow reconstruction of the sample collection and handling procedures at a later time.

A completed sample label will be attached to each investigative or QC sample. The following will be recorded with permanent ink on sample labels by the field sampling team:

- **Project name and number**
- **Sample number identification**
- **Initials of sampler**
- **Sampling location**
- **Required analysis**
- **Date and time of sample collection**
- **Space for laboratory sample number**
- **Preservative used, if applicable.**

The sample identification system will involve the following nomenclature "MWA-BBBB-CC" where:
"PSA" will denote

- **PS#- Monitoring Well Location**

"BBBB" will denote

- **MMYY – Month and year of sampling quarter, e.g.: First quarter (October), first year (2005), 1005**

"CC" will denote QA/QC sample

- **EB- equipment blank**
- **AD- analytical duplicate**
- **MS or MD – Matrix Spike or Matrix Duplicate**
- **TB- Trip Blank.**

Samples will be placed on ice inside a cooler immediately following sampling.

Chain-of-custody procedures will be instituted and followed throughout the sampling activities. Samples are physical evidence and will be handled according to strict chain-of-custody protocols. The field sampler is personally responsible for the care and custody of the sample until transferred. For proper identification in the field and proper tracking by the analytical laboratory, samples will be labeled in a clear and consistent fashion.

Field personnel will record the following information with permanent ink on the chain-of-custody:

- **Project identification and number**
- **Sample description/location**
- **Required analysis**
- **Date and time of sample collection**
- **Type and matrix of sample**
- **Number of sample containers**
- **Analysis requested/comments**
- **Sampler signature/date/time**
- **Air bill number.**

A chain-of-custody document, providing all information, signatures, dates, and other information, as required on the example chain-of-custody form included in SOP-9, will be completed by the field sampler and provided for each sample cooler.

Sampling containers will be packed in such a way as to help prevent breakage and cross-contamination. Samples will be shipped in coolers, each containing a chain-of-custody form and ice and ice packs to maintain inside temperature at approximately 4°C. Sample coolers will then be sealed between the lid and sides of the cooler with a custody seal prior to shipment. The custody seal will be an adhesive-

backed tape that easily rips if it is disturbed. Samples will be shipped to Severn-Trent Laboratories (STL) Savannah, Georgia for analysis by common overnight carrier.

Sample transportation will comply with U.S. Department of Transportation and IICAO/IATA (1999) regulations. Special sampling packing provisions will be made for samples requiring additional protection. Samples will remain in the custody of the sampler until transfer of custody is completed. Transfer consists of:

- **Delivery of samples to the laboratory sample custodian**
- **Signature of the laboratory sample custodian on the chain-of-custody document as receiving the samples, and signature of sampler, as relinquishing the samples.**

If a carrier is used to take samples between the sampler and the laboratory; a copy of the air bill must be attached to the chain-of-custody to maintain proof of custody.

When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the chain-of-custody. The field sampler will sign the chain-of-custody form when relinquishing custody, make a copy to keep with the field logbook, and include the original form in an air-tight plastic bag in the sample cooler with the associated samples.

The laboratory will assign a number for each sample upon receipt. That sample number will be placed on the sample label.

3.5 Field Documentation

URS personnel will keep a bound field notebook while performing sampling and oversight activities on-site. Forms that will be used include: chain-of-custody, test boring log, and field log, and sampling data sheets. The field logbooks will contain tabulated results of field measurements and documentation of field instrument calibration activities. The field logbooks will also record the following:

- **Personnel conducting the site activities, their arrival and departure times and their destination at the site**
- **Incidents and unusual activities that occur on the site such as, but not limited to, accidents, breaches of security, injuries, equipment failures, or weather related problems**
- **Changes to the FSP and the HASP**
- **Daily information such as:**
  - **Work accomplished and the current site status**
  - **Equipment calibrations, repairs and results**
  - **Site work zones.**
- **Date, time, weather conditions, equipment, and personnel on site**
- **Location where the work was performed**
- **Specific work activities conducted**
  - **Work zone and headspace readings.**

Entries will be signed and dated, and any entry which is to be deleted will have a single cross out which is signed and dated. The following sampling-related information will be recorded in the field logbook by the field sampling team:

- **Sample number**
- **Project identification**
- **Sampling location**
- **Required analysis**
- **Date and time of sample collection**
- **Type and matrix of sample**
- **Sampling technique**
- **Preservative used, if applicable**
- **Sampling conditions**
- **Observations**
- **Initials of the samplers**
- **Samples collected**
- **Water level**
- **Product level, if necessary**
- **Depth to bottom of well**

Photographic records will be developed through the use of digital photographs, showing pre-installation and post-installation of monitoring wells at each site.

Procedures to evaluate field data for this project primarily include checking for transcription errors on the part of field crew members and review of field notebooks. This task will be the responsibility of the URS Field Leader, who will otherwise not participate in making any of the field measurements or in adding notes, data, or other information to the notebook.

4.0 DATA MANAGEMENT, EVALUATION AND REPORTING

4.1 Data Management

The field data and documentation will become a part of the final file. The final file will be the central repository for all documents, which constitute evidence relevant to sampling and analysis activities as described in this plan. URS is the custodian of the file and maintains the contents of files for the site, including all relevant records, logs, field logbooks, pictures, subcontractor reports, data reviews, and the database management system.

The following documentation will supplement the chain-of-custody records:

- **Field logbooks and data**
- **Field collection report**
- **Photographs and drawings**
- **Contractor and subcontractor reports**
- **Correspondence.**

The file must be maintained in a secured, limited access area until all submittals for the project have been reviewed and approved, and for a minimum of six years past the submittal date of the final report. Upon completion of the analyses, URS will begin assimilating the field and laboratory notes. In this way, the file for the samples will be generated. The final file for the samples will be stored at URS and will consist of the following:

- **Laboratory data packages, including summary and raw data from the analysis of environmental and QC samples, chromatograms, mass spectra, calibration data, work sheets, and sample preparation notebooks**
- **Chain-of-custody records**
- **Data validation reports.**

Analytical data will be provided in hard copy and electronic formats. Electronic data will be loaded into a database to facilitate data evaluation and reporting. The data presented in the report will include the data flags provided by STL as well as the qualifiers assigned by URS.

4.2 Data Quality Objectives

The general objective of quality assurance is to collect defensible environmental data of known quality that is adequate for the data's intended use. To accomplish this, data quality objectives (DQOs) were developed. DQOs are qualitative and quantitative statements which clarify the study objective, define the most appropriate types of data to collect, determine the most appropriate conditions from which to collect data, and specify acceptable decisions regarding the data's usage (including reporting limits) to ensure that the collected data will fulfill the project objectives (U.S. EPA 1994).

Detected analyte concentrations will be compared to relevant Illinois Environmental Protection Agency (IEPA) Tiered Approach to Corrective Action Objectives guidelines. Table 4.1 lists the constituents and DQOs. URS will work with STL to attain the lowest reporting limits to meet the project objectives. However, due to technical constraints, achieving reporting limits that are lower than the DQOs might not be feasible for all compounds.

4.3 Quality Assurance Objectives

Quality assurance objectives for measurement of data are usually expressed in terms of precision, accuracy, completeness, representativeness, and comparability. The investigation will not be considered invalid if these criteria are not fully achieved but variances will trigger QA/QC measures to evaluate, and correct if necessary, any problem areas. The control limits for precision and accuracy as well as detection limits for each laboratory analysis are listed in Table 4.2.

Precision is a measure of the degree to which two or more measurements are in agreement. Field precision is assessed through the collection and measurement of field duplicates at a rate of one duplicate per ten analytical samples. Precision in the laboratory is assessed through the calculation of relative percent differences (RPD) for two or more replicate samples. The equations to be used for precision in this plan are presented in the model QAPP Section 14.2. The precision objective for laboratory analysis will be ± 25 percent RPD between field duplicates.

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy in the field is assessed through the use of field and trip blanks and through the adherence to all sample handling, preservation, and holding times. Laboratory accuracy is assessed through the analysis of matrix spikes (MS) or laboratory control samples (LCSs), and the determination of percent recoveries. The equation to be used for accuracy in this plan is presented in the model QAPP Section 14.1.

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. Field completeness for this project will be at least 90 percent. Laboratory completeness is a measure of the amount of valid measurements obtained from all the laboratory measurements taken in the project. The equation for completeness is presented in the model QAPP Section 14.3. Laboratory completeness for this project will be at least 95 percent. Results assigned an "R" qualifier would be unusable.

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is dependent upon the proper design of the sampling

program and will be satisfied by ensuring that the plan is followed and that proper sampling techniques are used. Representativeness in the laboratory data is ensured by using the proper analytical procedures, meeting sample holding times, and analyzing and assessing the field duplicate samples and matrix spike duplicate samples. The sampling network is designed to provide data representative of site conditions. During development of this network, consideration is given to existing analytical data, past site practices, and physical setting and processes.

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability can be related to precision and accuracy since these quantities are measures of data reliability. Samples from the same media are considered comparable if the procedures for collecting the samples are complied with and if the units of measurement are the same. Comparability is assured through the use of a laboratory for this project that uses established and approved analytical methods, protocols, and a laboratory quality control program designed to establish consistency in the performance of the analytical process. All data will be subjected to strict QA/QC procedures and reported in a consistent manner to allow for comparison across data sets.

4.4 Data Validation

Data validation will be performed by the URS QA Manager in accordance with QA/QC criteria established in the USEPA Region 5 Model QAPP. Current STL laboratory control limits and detection limits are included in Table 4.2. Excursions from QA/QC criteria will be qualified based on guidance provided in the following documents where applicable to the reference methods:

- **USEPA Contract Laboratory Program National Functional Guidelines for Low Concentration Organic Data Review. USEPA 540/R-94/012 (USEPA, 2001)**
- **USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. USEPA 540/R-94/013 (USEPA, 2004)**

A Level III validation will be performed for all data, and a Level IV validation will be performed for at least 10 percent of the data. Data validators will recalculate approximately 10 percent of the laboratory sample calculations using raw data when verifying sample results. In addition, data validators will review approximately 10 percent of the raw data to verify that compound identification was performed correctly and transcription errors are not present.

Data quality will be evaluated using laboratory or method control limits. Any control limits outside of the acceptable range shall be identified and reported. Sample data will be qualified based on excursions from laboratory or method control limits. Then, data validators will check corrective actions and results of reanalysis and document these events in the validation report.

Minor deficiencies in the data generation process noted in the data validation will result in approximation of sample data. Approximation of a data point indicates uncertainty in the reported concentration of the chemical but not its assigned identity. Major deficiencies noted in the data, validation will result in the rejection of sample results. Rejected data would be considered unusable for quantitative or qualitative purposes. Data qualifiers may include the following:

U **Indicates that the compound was analyzed for, but was not detected. The sample quantitation limit is presented and adjusted for dilution and percent moisture. This qualifier is also used to signify that the detection limit of an analyte was raised as a result of analytes detected in laboratory and/or field blank samples.**

J **Indicates that the detected sample result should be considered approximate based on excursions from QA/QC criteria.**

UJ **Indicates that the detection limit for the analyte in this sample should be considered approximate based on excursions from QA/QC criteria.**

R **Indicates that the previously reported detection limit or sample result has been rejected due to a major excursion from QA/QC criteria, for example percent recoveries of less than ten percent. The data should not be used for qualitative or quantitative purposes.**

The following method specific QA/QC parameters will be evaluated (at a minimum) during the data validation, where applicable.

Analyses for VOCs and SVOCs (where applicable)

- **Holding times and sample preservation**
- **Dilutions**
- **GC/MS instrument performance (Level IV validation only)**
- **Initial and continuing calibration (Level IV validation only)**
- **Blank analysis**
- **Surrogate recovery**
- **MS/MSD analysis**
- **Field duplicate analysis**
- **Laboratory Control Sample (LCS) analysis**
- **Internal standards performance**
- **Compound identification and quantification (Level IV validation only)**
- **Documentation completeness**
- **Overall assessment.**

Analyses for Pesticides and PCBs, (where applicable):

- **Holding times and sample preservation**
- **Dilutions**

- Initial and continuing calibration (Level IV validation only)
- Blank analysis
- Surrogate recovery
- MS/MSD analysis
- Field duplicate analysis
- Laboratory Control Sample (LCS) analysis
- Compound identification and quantification (Level IV validation only)
- Documentation completeness
- Overall assessment

Analyses for Herbicides, (where applicable):

- Holding times and sample preservation
- Dilutions
- Initial and continuing calibration (Level IV validation only)
- Blank analysis
- Surrogate recovery
- MS/MSD analysis
- Field duplicate analysis
- Laboratory Control Sample (LCS) analysis
- Compound identification and quantification (Level IV validation only)
- Documentation completeness
- Overall assessment.

Analyses for Metals, (where applicable):

- Holding times and sample preservation
- Dilutions
- Calibration (Level IV validation only)
- Blank analysis
- Spike analysis
- Field duplicate analysis
- Laboratory Control Sample (LCS) analysis
- Sample Result Verification (Level IV validation only)
- ICP Interference Check Samples (Level IV validation only)
- Laboratory Duplicates
- ICP Serial Dilution
- Documentation completeness
- Overall assessment

The laboratory will be conducting analyses on samples in accordance with methods listed in Table 4.3 and the laboratory's SOPs. The data will be provided to URS in an electronic format that will be uploaded into a database to facilitate data review and evaluation. The report tables will be generated from the

database. These tables will include the data flags provided by Savannah Labs as well as the data validation qualifiers assigned by URS.

Laboratory provided results (Form 1's) will also be included in the reports with the transcribed data qualifiers.

4.5 Quarterly Reporting

A groundwater monitoring report will be prepared and submitted to the USEPA after each sampling round when sample analysis and data validation are completed. The report will primarily include a discussion of sample collection procedures, groundwater elevation contour maps, a summary of the validated laboratory analytical data, and copies of the laboratory data. Groundwater elevation data from the monitoring wells and piezometers will be compiled by zone (i.e., SHU, MHU and DHU) and used to develop elevation contour maps.

4.6 Baseline Reporting

A baseline report will be prepared at the completion of two years of sampling. Concentration versus time plots will be created for each monitoring well in order to depict temporal changes in the concentration of key constituents. The monitored natural attenuation (MNA) parameters including alkalinity, carbon dioxide, chloride, ferrous iron, methane, nitrate, sulfate, total organic carbon will be analyzed and evaluated to assess the extent to which attenuation is occurring.

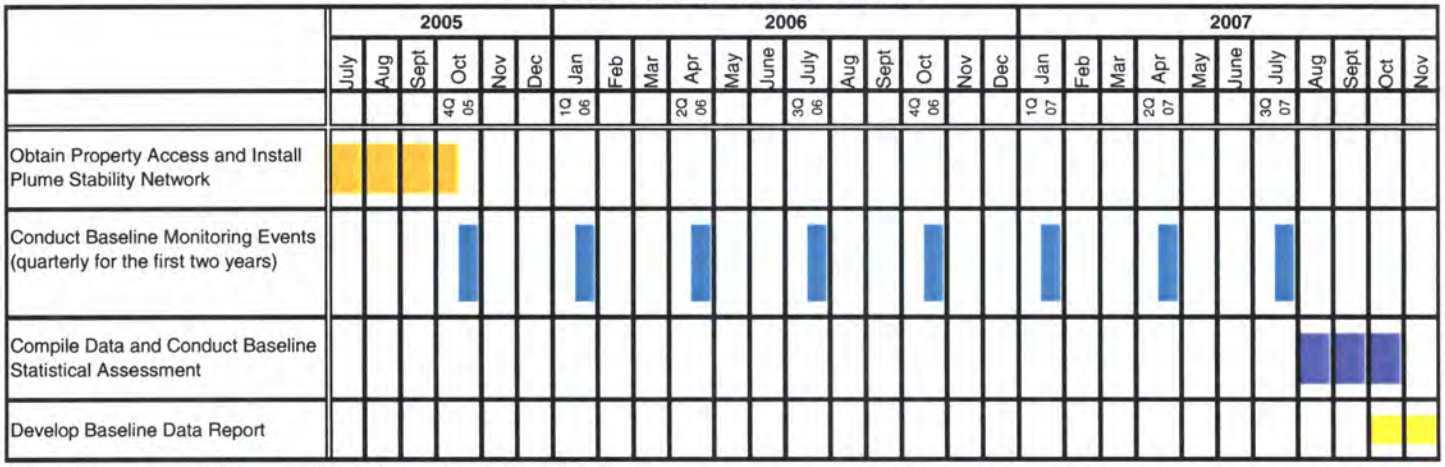
Analytical data from the baseline data collection period will be used to establish baseline statistical information such as normality, distribution, standard deviation, etc. The data distribution will be evaluated to determine if the data set is either normal, log normal or non-parametric. The baseline report will outline the statistical test or tests that will be used to determine the ongoing stability of the plume.

Groundwater, surface water and sediment data collected to monitor the Sauget Area 2 Groundwater Migration Control System will be considered, where applicable, in the evaluation of plume stability.

5.0 SCHEDULE

The schedule for the groundwater monitoring activities outlined in this work plan is approximately 29 months, as shown below. This includes securing property access, installing the plume stability network, conducting two years of quarterly monitoring information, and developing the statistical baseline.

SCHEDULE - GROUNDWATER MONITORING



Note: The baseline monitoring events include sampling, analysis and reporting.

6.0 REFERENCES

U.S. Environmental Protection Agency (USEPA) Region 5, 1998, *RCRA QAPP Instructions*, April 1998, Chicago, IL.

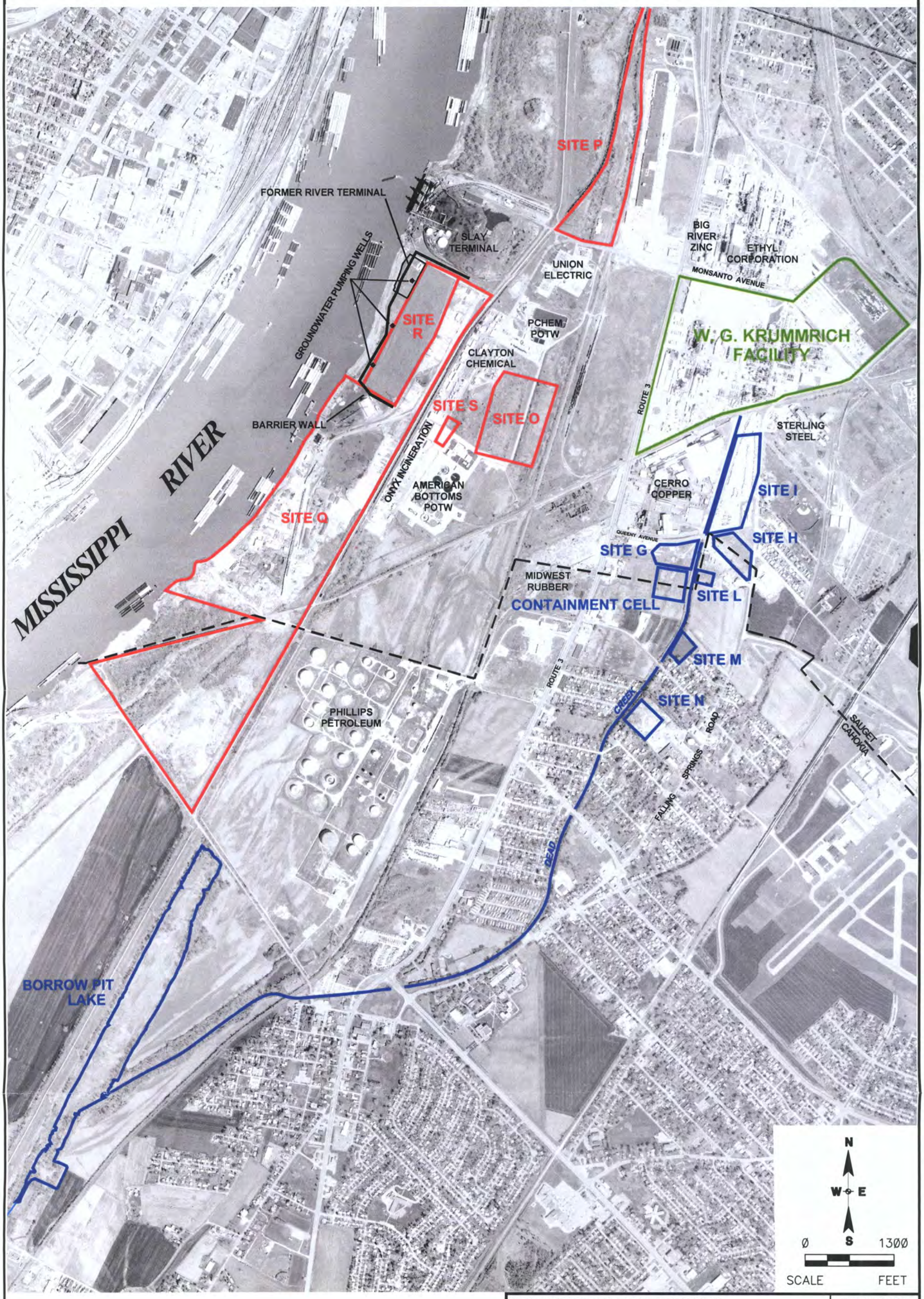
URS Corporation, 2004. *RCRA Corrective Measures Study (CMS), Solutia Inc., W.G. Krummrich Facility, Sauget, Illinois.*

URS Corporation, 2005. *RCRA Corrective Measures Study (CMS), Response to Comments, Solutia Inc., W.G. Krummrich Facility, Sauget, Illinois.*

URS Corporation, 2005. *RCRA Corrective Measures Study, Response to May 4, 2005 USEPA Comments, Solutia Inc., W.G. Krummrich Facility Investigation, Sauget, Illinois.*

FIGURES

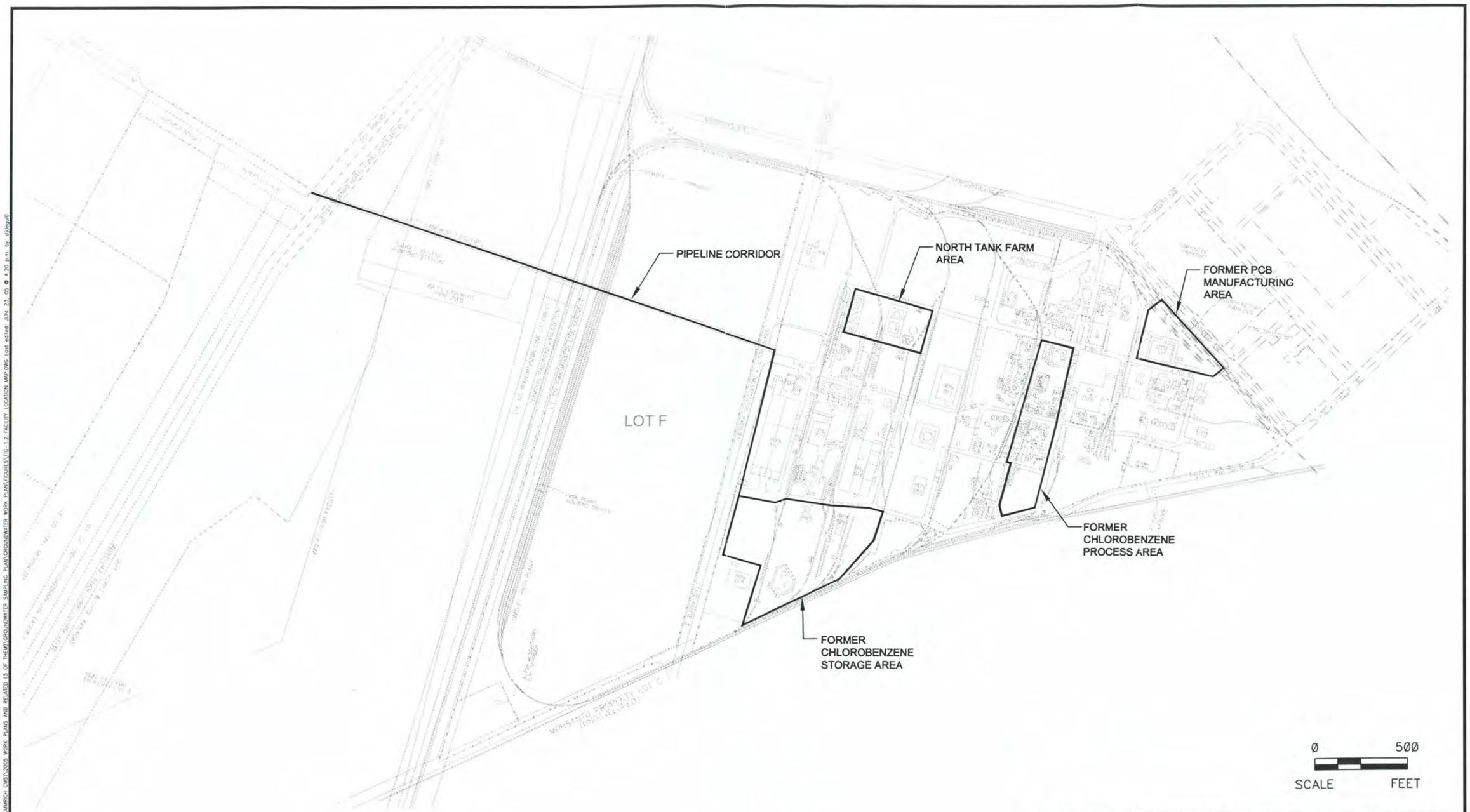
File: P:\ENVIRONMENTAL\21561388 (SOLUTIA KRUMMRICH CMS)\2005 WORK PLANS AND RELATED (3 OF THEM)\GROUNDWATER SAMPLING PLAN\GROUNDWATER WORK PLAN\FIGURES\FIG-1.1 SITE LOCATION MAP.DWG Last edited: JUN. 23, 05 @ 12:53 p.m. by: djdequi0



LEGEND

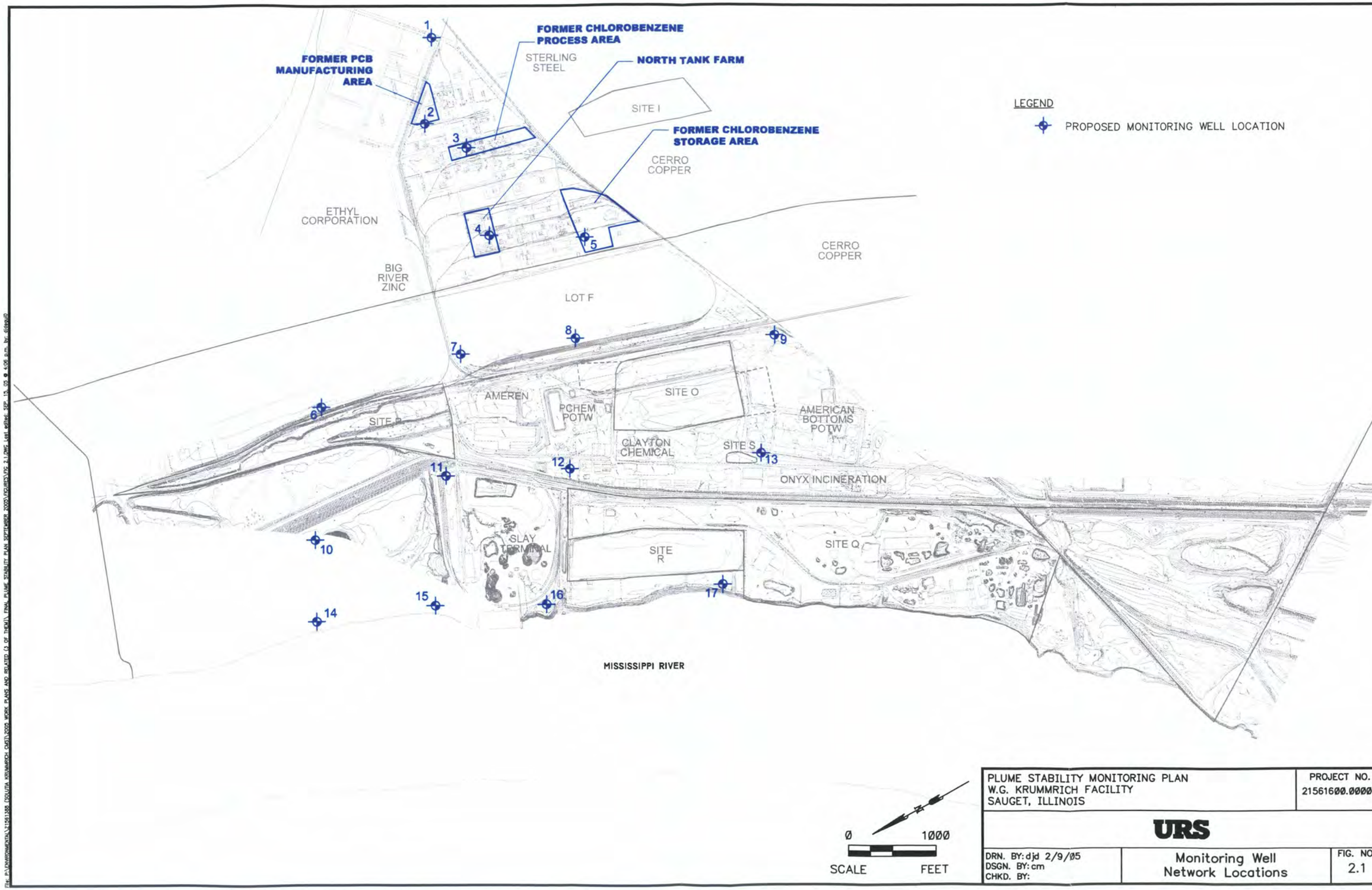
- W.G. KRUMMRICH FACILITY
- SAUGET AREA #1
- SAUGET AREA #2

PLUME STABILITY MONITORING PLAN W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS		PROJECT NO. 21561573.00000
URS		
DRN. BY: 2/9/05 DSGN. BY: tja CHKD. BY:	Site Location Map	FIG. NO. 1.1



PLUME STABILITY MONITORING PLAN W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS	PROJECT NO. 21561573.00000
URS	
DRN. BY: 4/9/05 DSGN. BY: cm CHKD. BY:	Facility Location Map — FIG. NO. 1.2

File: P:\ENVIRONMENTAL\21561368 (SOLUTIA KRUMMRICH CMS)\2005 WORK PLANS AND RELATED (3 OF THEM)\GROUNDWATER SAMPLING PLAN\GROUNDWATER WORK PLAN\FIGURES\FIG-1.2 FACILITY LOCATION MAP.DWG Last edited JUN. 23, 05 @ 4:20 p.m. by djdegu0



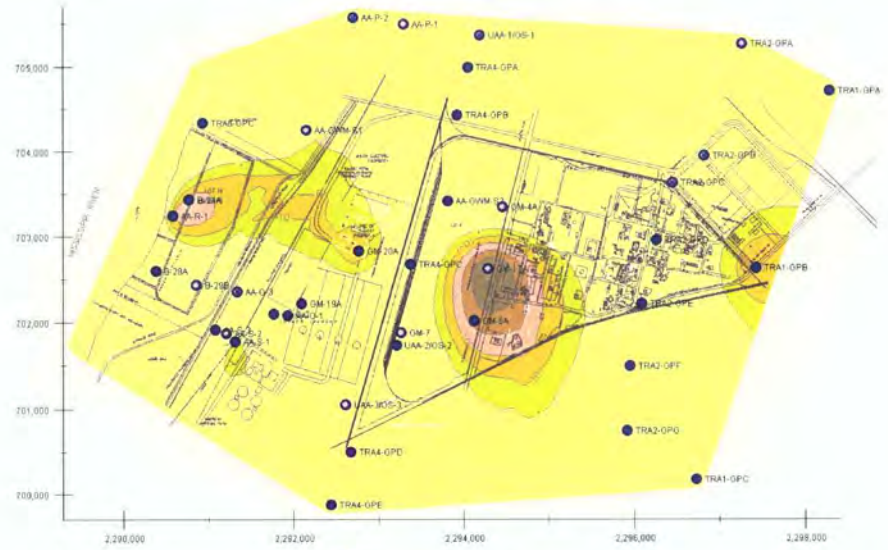
PLUME STABILITY MONITORING PLAN W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS	PROJECT NO. 21561600.00000
URS	
DRN. BY: djd 2/9/05 DSGN. BY: cm CHKD. BY:	Monitoring Well Network Locations	FIG. NO. 2.1

Distribution of Benzene in Groundwater

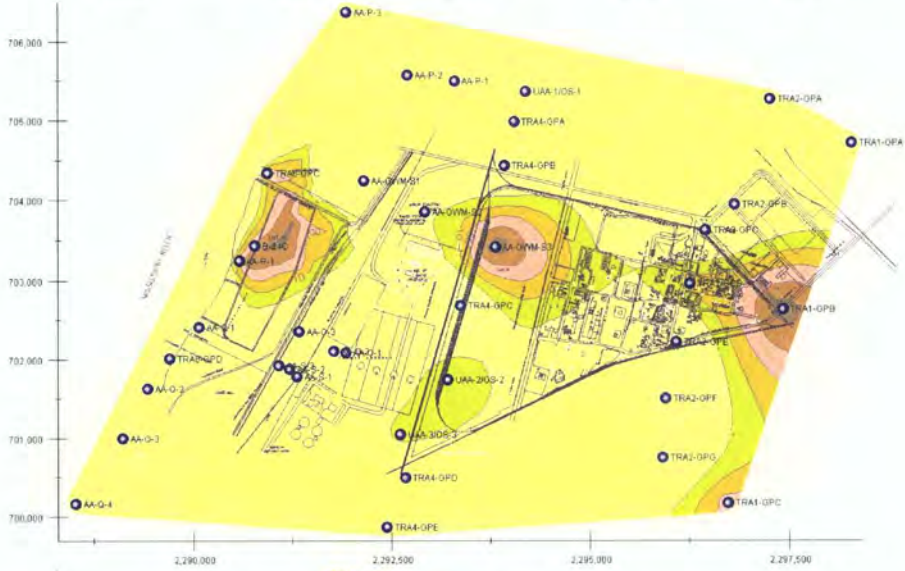
URS

N

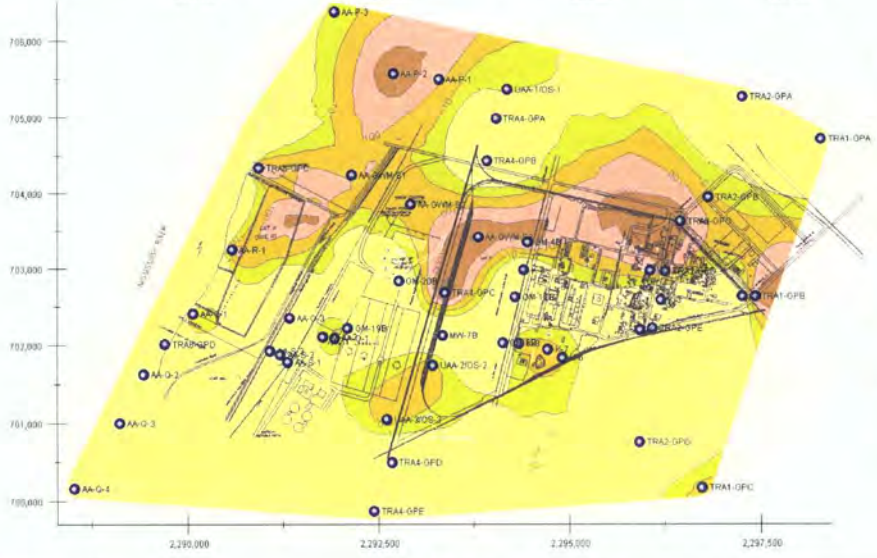
SHU
(Elevation
395 to 380)



MHU
(Elevation
380 to 350)



DHU
(Elevation 350
to Bedrock)



- <1 ug/l
- 1 to 10 ug/l
- 10 to 100 ug/l
- 100 to 1000 ug/l
- 1000 to 10000 ug/l
- >10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: 2.2

Project Number: 21561601

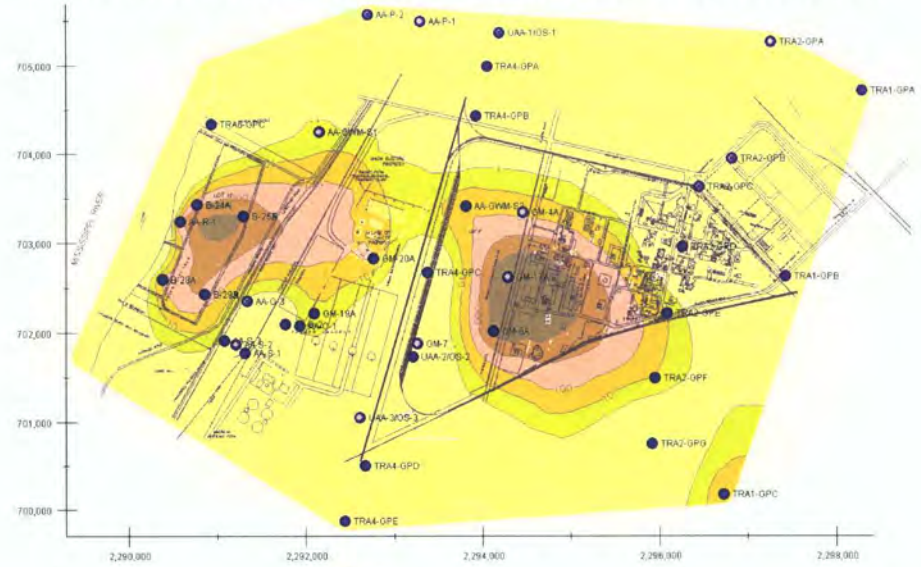
Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Distribution of Chlorobenzene in Groundwater

URS

N

SHU
(Elevation
395 to 380)



MHU
(Elevation
380 to 350)

DHU
(Elevation 350
to Bedrock)

- <1 ug/l
- 1 to 10 ug/l
- 10 to 100 ug/l
- 100 to 1000 ug/l
- 1000 to 10000 ug/l
- >10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: 2.3

Project Number: 21561601

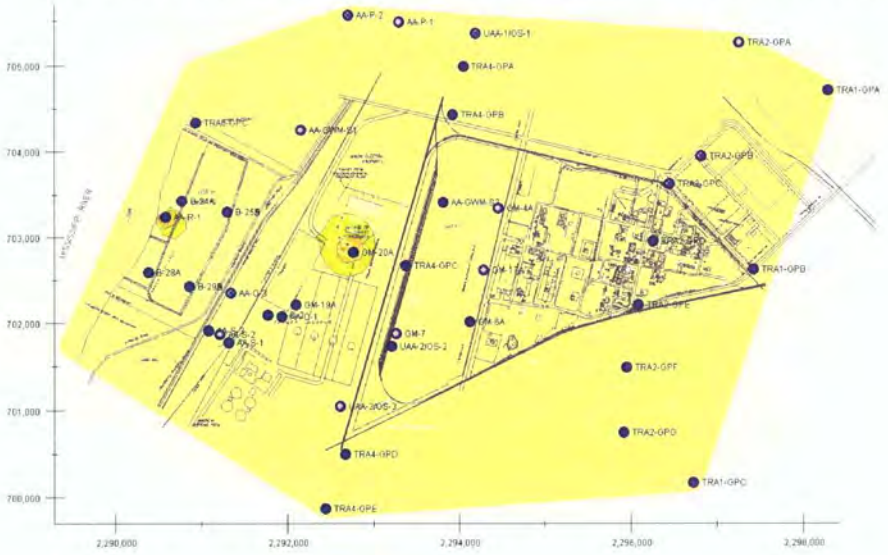
Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Distribution of Chloroanilines in Groundwater

URS

N

SHU
(Elevation
395 to 380)



MHU
(Elevation
380 to 350)

DHU
(Elevation 350
to Bedrock)

- <1 ug/l
- 1 to 10 ug/l
- 10 to 100 ug/l
- 100 to 1000 ug/l
- 1000 to 10000 ug/l
- >10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: 2.4

Project Number: 21561601

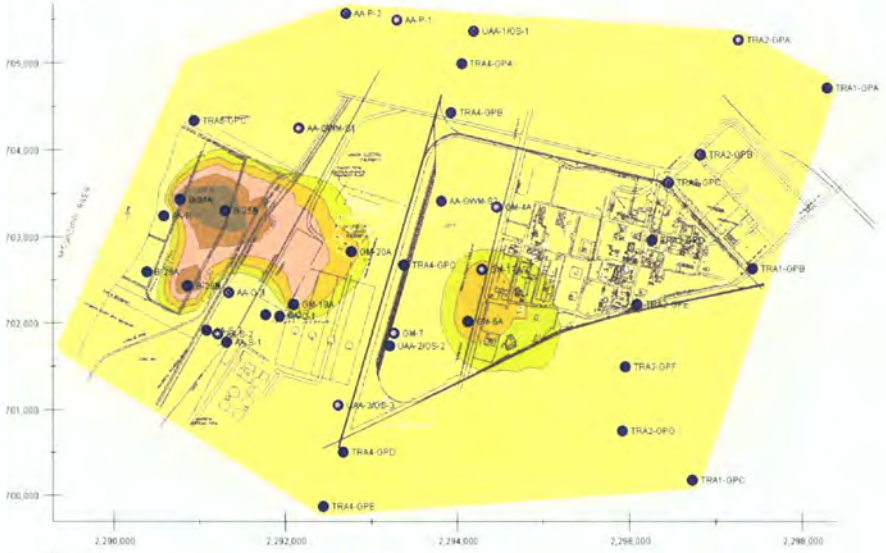
Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Distribution of Chlorophenols in Groundwater

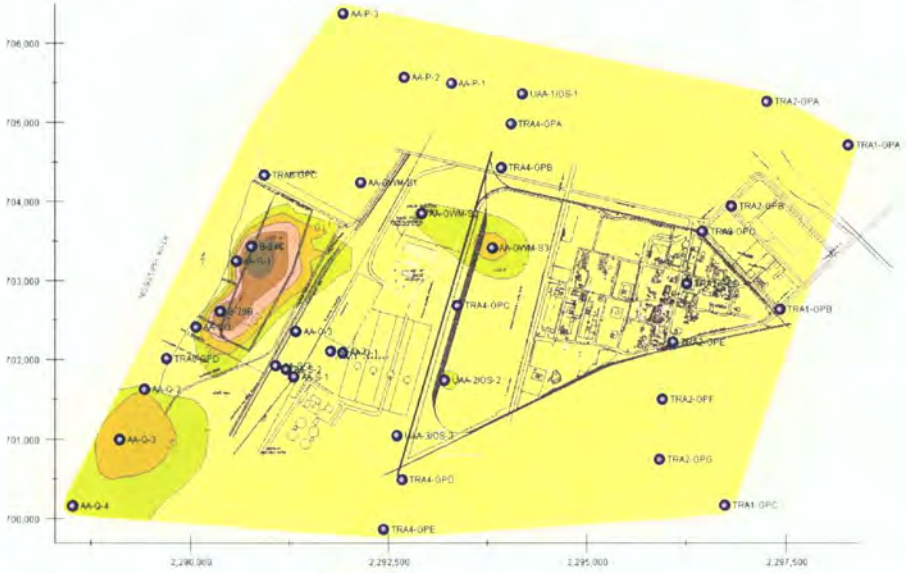
URS

N

SHU
(Elevation
395 to 380)



MHU
(Elevation
380 to 350)



DHU
(Elevation 350
to Bedrock)

- <1 ug/l
- 1 to 10 ug/l
- 10 to 100 ug/l
- 100 to 1000 ug/l
- 1000 to 10000 ug/l
- >10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: 2.5

Project Number: 21561601

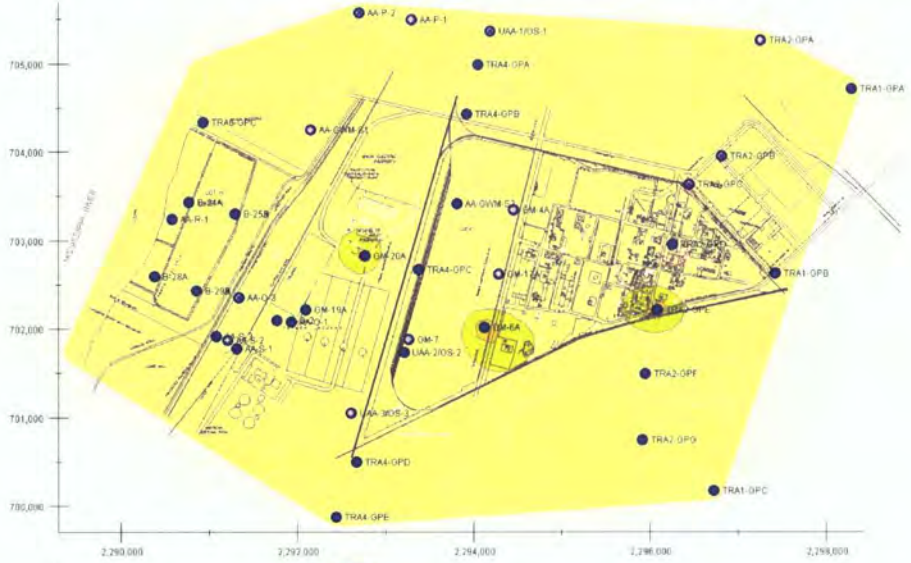
Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Distribution of Dichlorobenzenes in Groundwater

URS

N

SHU
(Elevation
395 to 380)



MHU
(Elevation
380 to 350)

DHU
(Elevation 350
to Bedrock)

- <1 ug/l
- 1 to 10 ug/l
- 10 to 100 ug/l
- 100 to 1000 ug/l
- 1000 to 10000 ug/l
- >10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: 2.6

Project Number: 21561601

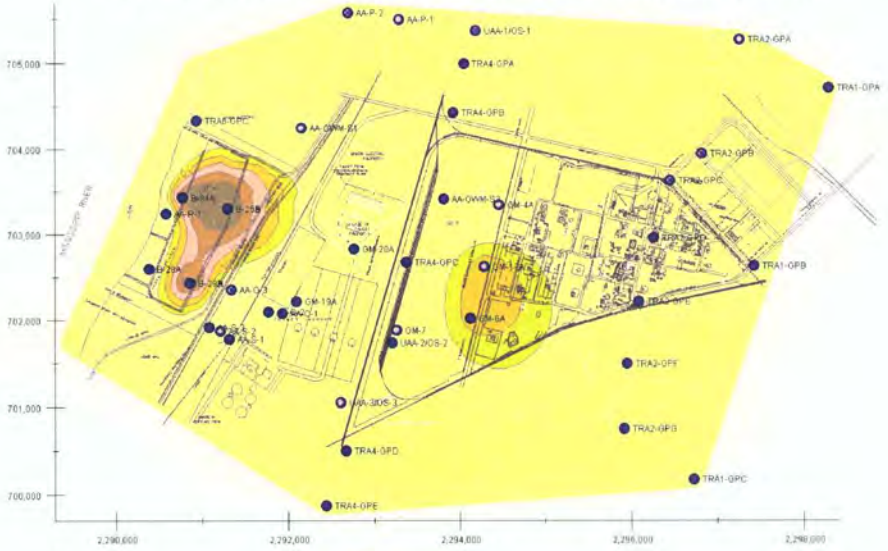
Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Distribution of Phenol in Groundwater

URS

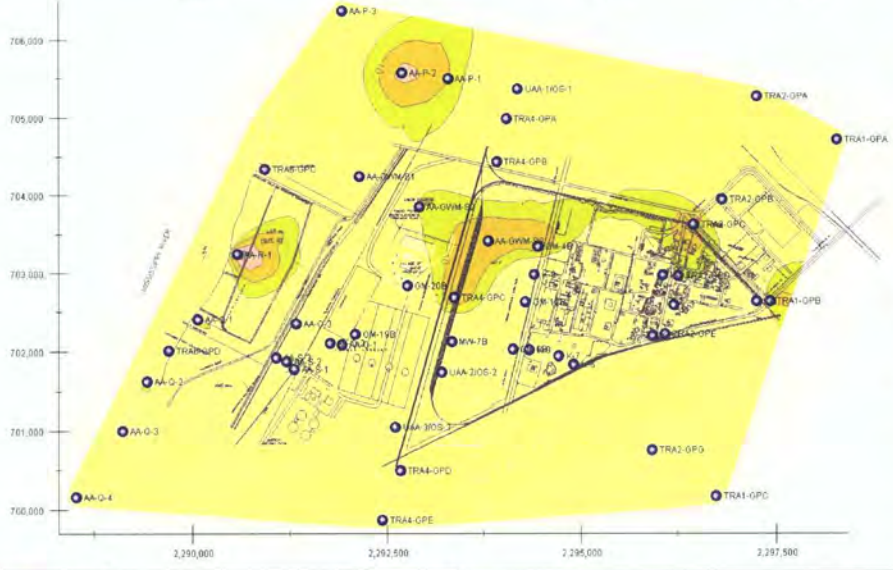
N

SHU
(Elevation
395 to 380)



MHU
(Elevation
380 to 350)

DHU
(Elevation 350
to Bedrock)



- <1 ug/l
- 1 to 10 ug/l
- 10 to 100 ug/l
- 100 to 1000 ug/l
- 1000 to 10000 ug/l
- >10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: 2.7

Project Number: 21561601

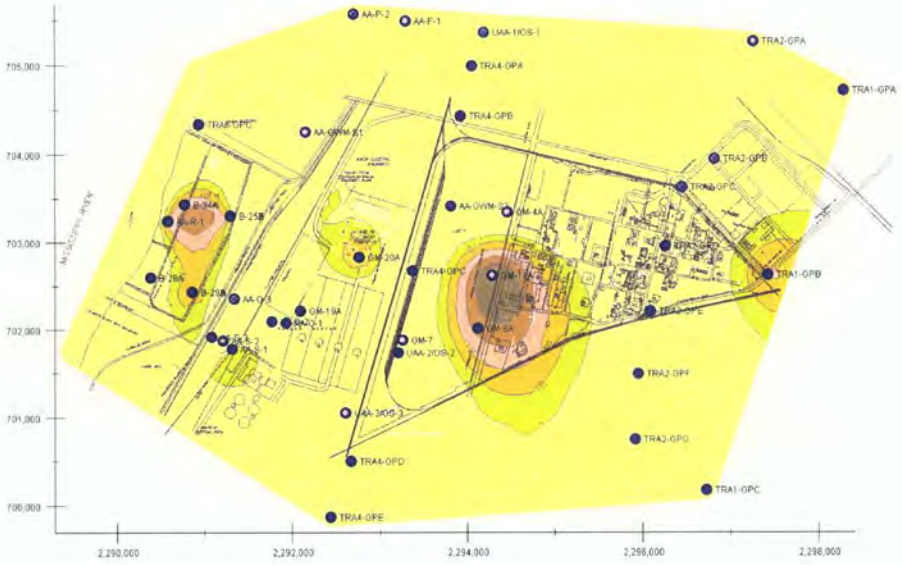
Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Distribution of BTEX in Groundwater

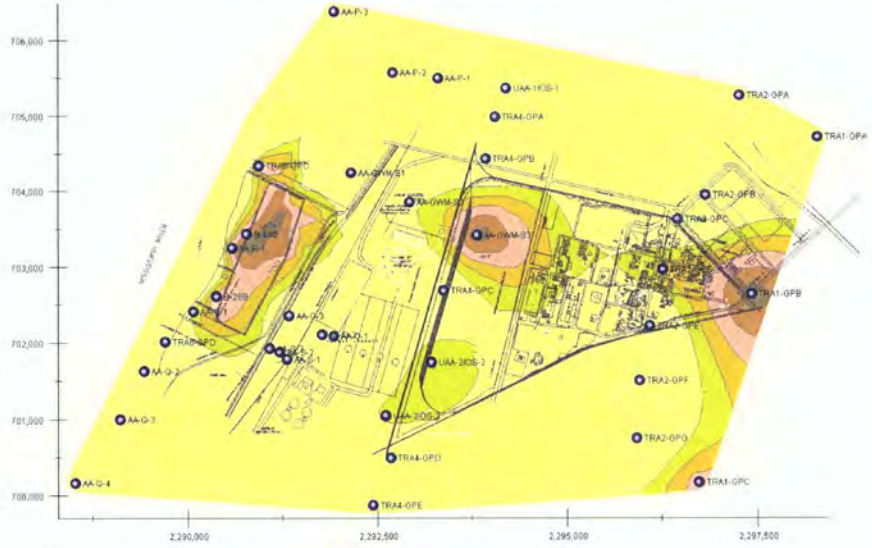
URS

N

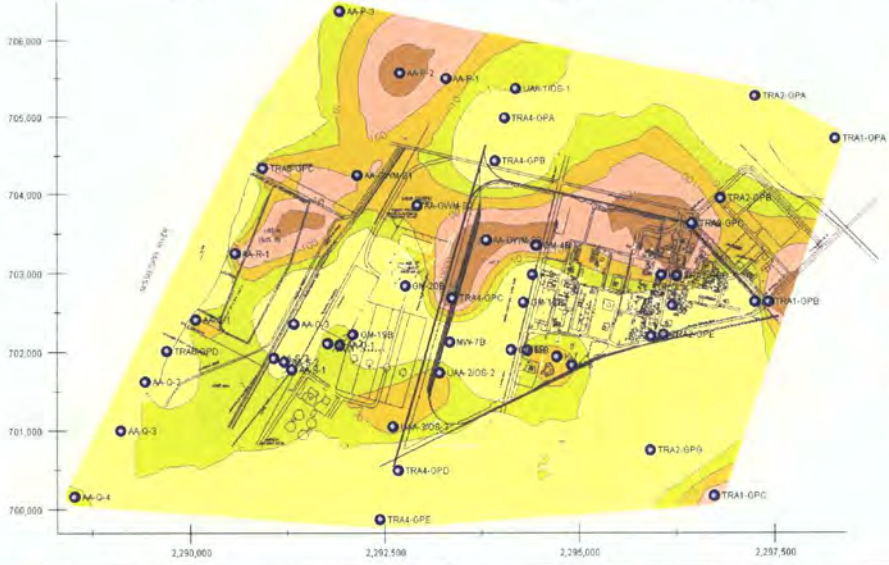
SHU
(Elevation
395 to 380)



MHU
(Elevation
380 to 350)



DHU
(Elevation 350
to Bedrock)



- <1 ug/l
- 1 to 10 ug/l
- 10 to 100 ug/l
- 100 to 1000 ug/l
- 1000 to 10000 ug/l
- >10000 ug/l

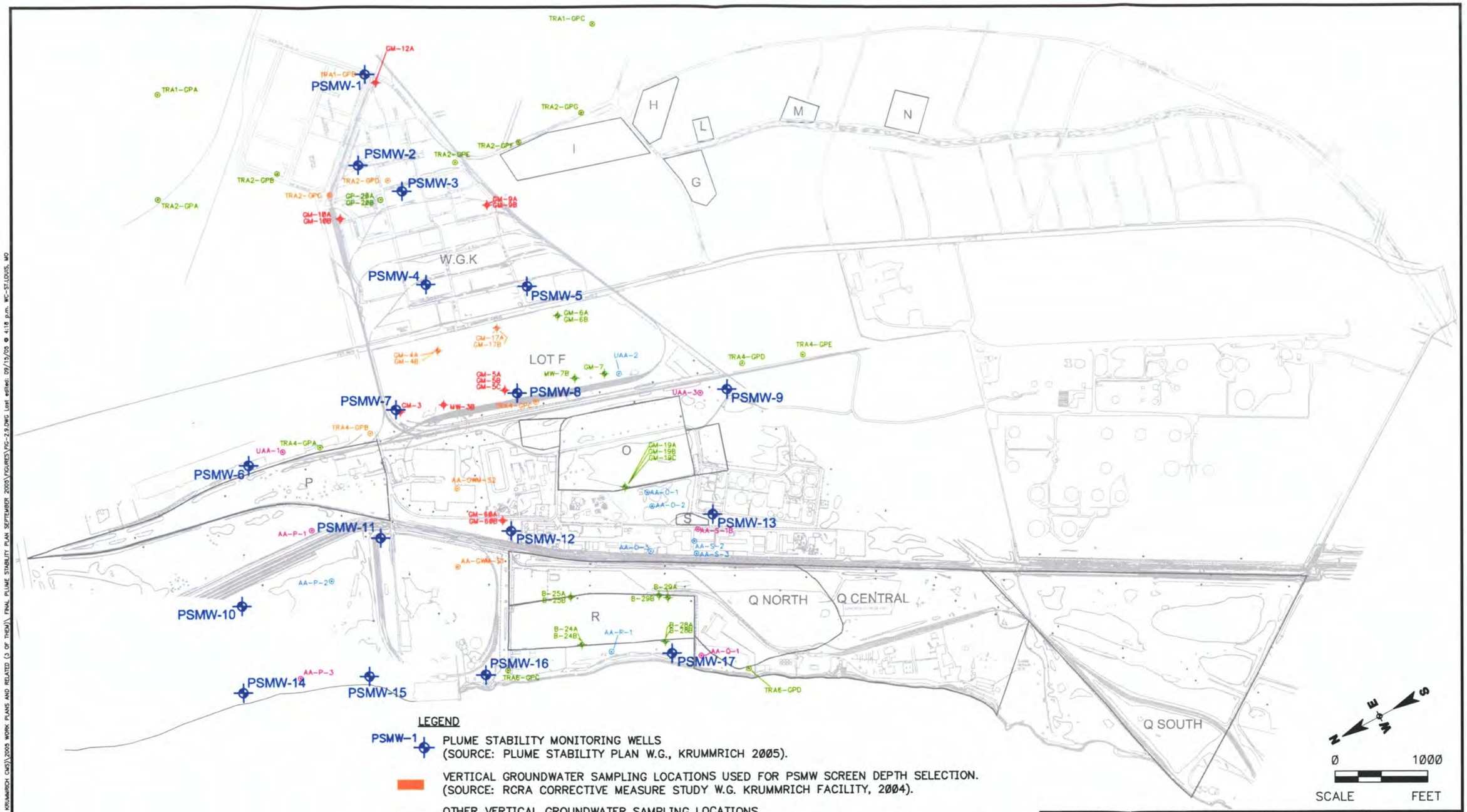
SOLUTIA

Date: 07/14/2005

Figure Number: 2.8

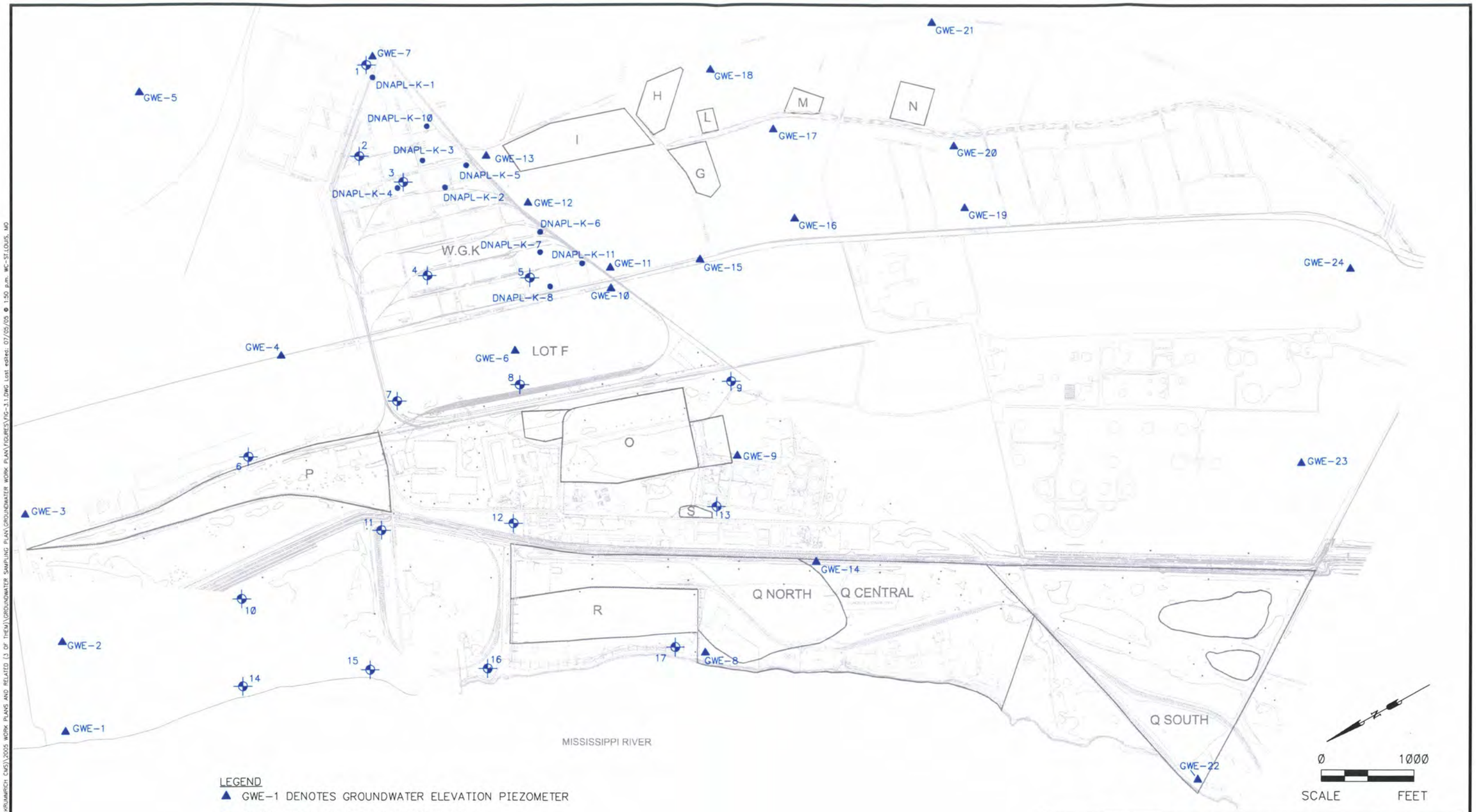
Project Number: 21561601

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois



File: P:\ENVIRONMENTAL\21561388 (SOLUTIA KRUMMRICH CMS)\2005 WORK PLANS AND RELATED (3 OF THEM)\ FINAL PLUME STABILITY PLAN SEPTEMBER 2005\FIGURES\FIG-2.9.DWG Last edited: 09/19/05 @ 4:18 p.m. WC-ST.LOUIS, MO

W.G. KRUMMRICH FACILITY, SAUGET, ILLINOIS PLUME STABILITY MONITORING PLAN		PROJECT NO. 21561600.00000
URS		
DRN. BY:djd 8/9/05 DSGN. BY: jp CHKD. BY:	Vertical Groundwater Profile Locations	FIG. NO. 2.9



LEGEND

- ▲ GWE-1 DENOTES GROUNDWATER ELEVATION PIEZOMETER
- • DNAPL GROUNDWATER MONITORING WELL
- ⊕ PLUME STABILITY MONITORING WELL

NOTE:
EACH PIEZOMETER LOCATION INCLUDES A CLUSTER OF THREE GROUNDWATER ELEVATION PIEZOMETERS.

PLUME STABILITY MONITORING PLAN W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS	PROJECT NO. 21561573.ØØØØØ
URS	
DRN. BY: djd 2/9/Ø5 DSGN. BY: cm CHKD. BY:	Water Level Gauging Locations
	FIG. NO. 3.1

File: P:\ENVIRONMENTAL\21561388 (SOLUTIA KRUMMRICH CMS)\2005 WORK PLANS AND RELATED (3 OF THEM)\GROUNDWATER SAMPLING PLAN\GROUNDWATER WORK PLAN\FIGURES\FIG-3.1.DWG Last edited: 07/05/05 @ 1:50 p.m. WC-ST.LOUIS, MO

TABLES

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 1
Table 2.1

	Sample Depth		
VOCs, ug/l	21 (ft bgs)	43 (ft bgs)	73 (ft bgs)
Benzene	150	**37,000**	7,800
Ethylbenzene	ND(5)	**1,900**	370
Toluene	ND(5)	**520**	250
Total Xylenes	24	**5,000**	530
Total VOCs	174	**44,420**	8,950
SVOCs, ug/l			
2,4-Dimethylphenol	ND(10)	**120**	ND(10)
2-Methylnaphthalene	39	**79**	58
Naphthalene	ND(10)	**270**	11
Phenol	ND(10)	**81**	26
Total SVOCs		**550**	95

Notes:

1) Data Source: W.G. Krummrich Facility CMS Data Report
2) Closest Vertical Groundwater Profile: TRA1-GPB
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: July 2003
6) Selected Screen Depth: 43 ft bgs

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 2
Table 2.2

	Sample Depth		
VOCs, ug/l	**25 (ft bgs)**	**43 (ft bgs)**	**73 (ft bgs)**
Chlorobenzene	ND(5)	120	**1,200**
Total VOCs		120	**1,200**
SVOCs, ug/l			
1,4-Dichlorobenzene	ND(10)	**14**	ND(10)
2-Chlorophenol	ND(10)	ND(10)	**10**
4-Chloroaniline	ND(20)	ND(20)	**91**
Total SVOCs		14	**101**

Notes:

1) **Data Source: W.G. Krummrich Facility CMS Data Report**
2) **Closest Vertical Groundwater Profile: TRA2-GPC**
3) **Highest Detected Concentrations in Bold**
4) **Concentrations < 1 ug/l Not Reported**
5) **Sample Collection Date: June 2003**
6) **Selected Screen Depth: 73 ft bgs**

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 3
Table 2.3

	Sample Depth		
VOCs, ug/l	21 (ft bgs)	(ft bgs)	72 (ft bgs)
Benzene	ND(5)	49	**1,300**
Chlorobenzene	ND(5)	**1,800**	1,200
Ethylbenzene	ND(5)	ND(25)	**240**
Toluene	ND(5)	ND(25)	**99**
Total VOCs		1,849	**2,839**
SVOCs, ug/l			
1,2-Dichlorobenzene	ND(10)	ND(10)	**230**
1,3-Dichlorobenzene	ND(10)	**40**	31
1,4-Dichlorobenzene	ND(10)	**920**	570
2,4-Dimethylphenol	ND(10)	ND(10)	**64**
2-Methylnaphthalene	ND(10)	ND(10)	**39**
3 & 4-Methylphenol	ND(10)	ND(10)	**33**
4-Chloroaniline	ND(20)	**33**	31
Naphthalene	ND(10)	ND(10)	**85**
Phenol	ND(10)	ND(10)	**11**
Total SVOCs		993	**1,094**
Total VOCs and SVOCs		2,842	**3,933**

Notes:

1) Data Source: W.G. Krummrich Facility CMS Data Report
2) Closest Vertical Groundwater Profile: TRA2-GPD
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: July 2003
6) Selected Screen Depth: 72 ft bgs

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 4
Table 2.4

	Sample Depth		
VOCs, ug/l	13 - 28 (ft bgs)	67 - 87 (ft bgs)	92.5 - 102.5 (ft bgs)
Benzene	ND(5)	2,800	**19,000**
Chlorobenzene	46	1,600	**9,600**
Total VOCs	46	4,400	**28,600**
SVOCs, ug/l			
2-Chlorophenol	ND(10)	10	**22**
4-Chloroaniline	ND(20)	44	**46**
Phenol	ND(10)	64	**170**
Total SVOCs		118	**238**
Total VOCs and SVOCs		4,518	**28,838**

Notes:

1) Data Source: W.G. Krummrich Facility CMS Data Report
2) Closest Vertical Groundwater Profile: GM-4 A, B and C
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: August 2003
6) Selected Screen Depth: 92.5 - 102.5 ft bgs

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 5
Table 2.5

	Sample Depth		
VOCs, ug/l	18 - 38 (ft bgs)	58 - 78 (ft bgs)	87 - 107 (ft bgs)
Benzene	**760,000**	120	ND(50)
Chlorobenzene	**220,000**	3,300	1,500
Total VOCs	**980,000**	3,420	1,500
SVOCs, ug/l			
1,2-Dichlorobenzene	ND(10)	**15**	12
1,3-Dichlorobenzene	ND(10)	ND(10)	**12**
1,4-Dichlorobenzene	ND(10)	130	**240**
2-Chlorophenol	**59**	17	ND(10)
Naphthalene	**15**	ND(10)	ND(10)
p-Chloroaniline	ND(20)	ND(20)	**24**
Phenol	**100**	ND(10)	ND(10)
Total SVOCs	174	162	**288**
Total VOCs and SVOCs	**980,174**	3,582	1,788

Notes:

1) Data Source: W.G. Krummrich Facility CMS Data Report
2) Closest Vertical Groundwater Profile: GM-17 A, B and C
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: August 2003
6) Selected Screen Depth: 18 - 38 ft bgs
7) Selected Screen Depth Adjusted to 20 to 25 ft bgs Based on Data from VOC GM-33, 34 & 35

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 6
Table 2.6

	Sample Depth									
VOCs, ug/l	20 (ft bgs)	30 (ft bgs)	40 (ft bgs)	50 (ft bgs)	60 (ft bgs)	70 (ft bgs)	80 (ft bgs)	90 (ft bgs)	100 (ft bgs)	110 (ft bgs)
1,2-Dichloroethene	ND(2)	ND(2)	ND(2)	0.82	1.3	**1.7**	1.4	1.5	1.3	ND(4)
Benzene	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(2)	0.23	2.5	**3.4**
Chlorobenzene	ND(1)	ND(1)	ND(1)	0.42	2	2.4	280	450	400	**710**
Total VOCs				1.24	3.3	4.1	281.4	451.73	403.8	**713.4**
SVOCs, ug/l										
1,2-Dichlorobenzene	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	1.4	3.8	**6.3**
1,4-Dichlorobenzene	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	1.8	9.8	18	**36**
2-Chlorophenol	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	3.3	5.4	1.7	**6.4**
Total SVOCs							5.1	16.6	23.5	**48.7**
Total VOCs and SVOCs				1.24	3.3	4.1	286.5	468.33	427.3	**762.1**

Notes:

1) Data Source: Sauget Area 2 RI/FS Data Report
2) Closest Vertical Groundwater Profile: UAA-1
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: July 2002
6) Selected Screen Depth: 110 ft bgs

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 7
Table 2.7

	Sample Depth			
VOCs, ug/l	29 (ft bgs)	49 (ft bgs)	76 (ft bgs)	119 (ft bgs)
Chlorobenzene	ND(5)	8.7	400	**8,300**
Total VOCs		8.7	400	**8,300**
SVOCs, ug/l				
1,4-Dichlorobenzene	ND(10)	ND(10)	ND(10)	**53**
2-Chlorophenol	ND(10)	ND(10)	ND(10)	**24**
4-Chloroaniline	ND(20)	ND(20)	ND(20)	**22**
Total SVOCs				**99**
Total VOCs and SVOCs		8.7	400	**8,399**

Notes:

1) Data Source: W.G. Krummrich Facility CMS Data Report
2) Closest Vertical Groundwater Profile: TRA4-GPB
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: July 2003
6) Selected Screen Depth: 119 ft bgs

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 8
Table 2.8

	Sample Depth			
VOCs, ug/l	21 (ft bgs)	43 (ft bgs)	73 (ft bgs)	119 (ft bgs)
Benzene	ND(5)	ND(5)	**160,000**	120
Chlorobenzene	ND(5)	ND(5)	**57,000**	3,200
Total VOCs			**217,000**	3,320
SVOCs, ug/l				
1,2Dichlorobenzene	ND(10)	ND(10)	ND(10)	11
1,3-Dichlorobenzene	ND(10)	ND(10)	ND(10)	11
1,4-Dichlorobenzene	ND(10)	ND(10)	33	**280**
2-Chlorophenol	ND(10)	ND(10)	22	**34**
4-Chloroaniline	ND(20)	ND(20)	**140**	ND(20)
Naphthalene	ND(10)	ND(10)	ND(10)	**22**
Phenol	ND(10)	ND(10)	**200**	ND(10)
Total SVOCs			**395**	**358**
Total VOCs and SVOCs			**217,395**	3,678

Notes:

1) Data Source: W.G. Krummrich Facility CMS Data Report
2) Closest Vertical Groundwater Profile: TRA4-GPC
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: June 2003
6) Selected Screen Depth: 73 ft bgs

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 9
Table 2.9

	Sample Depth										
VOCs, ug/l	24 (ft bgs)	34 (ft bgs)	44 (ft bgs)	54 (ft bgs)	64 (ft bgs)	74 (ft bgs)	84 (ft bgs)	94 (ft bgs)	104 (ft bgs)	114 (ft bgs)	116 (ft bgs)
1,2-Dichloroethene	ND(2)	ND(2)	1.3	1.4	1.3	**3.5**	ND(20)	ND(40)	ND(40)	ND(10)	ND(4)
Benzene	ND(1)	ND(1)	3.6	1.2	0.14	7.4	**63**	55	24	4.3	1.8
Chlorobenzene	ND(1)	ND(1)	160	160	31	150	1300	**2100**	**2100**	570	360
Total VOCs			165	163	32.44	160.9	1,363	**2155**	2124	574.3	361.8
SVOCs, ug/l											
1,2-Dichlorobenzene	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	5	17	**25**	23	16
1,3-Dichlorobenzene	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	20	**28**	18	13
1,4-Dichlorobenzene	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	170	1,000	1,700	**1,800**	1,500
2,4,6-Trichlorophenol	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	**7.8**	1	1.5
2,4-Dichlorophenol	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	2.2	9.4	52	**59**	53
2-Chlorophenol	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	14	9.5	14	**17**	9.7
Benzo(a)pyrene	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	**1.4**	ND(10)	ND(10)
Benzo(b)fluoranthene	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	**1.3**	ND(10)	ND(10)
Benzo(g,h,i)perylene	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	**1.6**	ND(10)	0.69
Dibenzo(a,h)anthracene	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	**1.5**	0.67	ND(10)
Diethyl Phthalate	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	17	**3.2**	ND(10)	ND(10)
4-Chloroaniline	ND(20)	ND(20)	ND(20)	ND(20)	ND(20)	ND(20)	470	800	**1,400**	ND(200)	9.8
p-Nitroaniline	ND(20)	ND(20)	ND(20)	ND(20)	ND(20)	ND(20)	ND(50)	ND(50)	**1.5**	ND(50)	ND(50)
Total SVOCs							661.2	1,873	**3,237**	1,919	1,604
Total VOCs and SVOCs							2,024	4,028	**5,361**	2,493	1,965

Notes:
1) Data Source: Sauget Area 2 RI/FS Data Report
2) Closest Vertical Groundwater Profile: UAA-3
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: June 2002
6) Selected Screen Depth: 104 ft bgs

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selections
Monitoring Wells 10 & 11
Table 2.10

	Sample Depth										
VOCs, ug/l	24 (ft bgs)	34 (ft bgs)	44 (ft bgs)	54 (ft bgs)	64 (ft bgs)	74 (ft bgs)	84 (ft bgs)	94 (ft bgs)	104 (ft bgs)	114 (ft bgs)	120 (ft bgs)
2-Butanone	7.5	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(250)	ND(250)	ND(100)
Benzene	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	**190**	60	77
Carbon Disulfide	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(25)	ND(25)	**6.4**
Chlorobenzene	ND(1)	ND(1)	ND(1)	ND(1)	1	5.8	12	4.8	**4,900**	4,600	2,300
Total VOCs	7.5				1	5.8	12	4.8	**5,090**	4,660	2,383
SVOCs, ug/l											
1,4-Dichlorobenzene	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	9.8	3.1
2-Chlorophenol	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	**78**	46	22
2-Nitroaniline	ND(50)	ND(50)	ND(50)	ND(50)	**1.2**	ND(50)	ND(50)	ND(50)	ND(100)	ND(50)	ND(50)
3 & 4-Methylphenol	ND(10)	ND(10)	ND(10)	ND(10)	**1.4**	ND(10)	ND(10)	ND(10)	ND(20)	ND(10)	ND(10)
4,6-Dinitro-2-methylphenol	ND(50)	ND(50)	ND(50)	ND(50)	**5.2**	ND(50)	ND(50)	ND(50)	ND(100)	ND(50)	ND(50)
Benzo(a)anthracene	ND(10)	ND(10)	ND(10)	ND(10)	**1**	ND(10)	ND(10)	ND(10)	ND(20)	ND(10)	ND(10)
Benzo(b)fluoranthene	ND(10)	ND(10)	ND(10)	ND(10)	**4**	ND(10)	ND(10)	ND(10)	ND(20)	ND(10)	ND(10)
Benzo(g,h,i)perylene	ND(10)	ND(10)	ND(10)	ND(10)	**1.1**	ND(10)	ND(10)	ND(10)	ND(20)	ND(10)	ND(10)
Bis-2-choroethyl ether	ND(10)	ND(10)	ND(10)	ND(10)	**1.1**	ND(10)	ND(10)	ND(10)	ND(20)	ND(10)	ND(10)
Bis(2-ethylhexyl)phthalate	ND(10)	ND(10)	ND(10)	ND(10)	**1.2**	ND(10)	ND(10)	ND(10)	ND(20)	ND(10)	ND(10)
Di-n-butyl Phthalate	ND(10)	ND(10)	ND(10)	ND(10)	**1.1**	ND(10)	ND(10)	ND(10)	ND(20)	ND(10)	ND(10)
Dibenzo(a,h)anthracene	ND(10)	ND(10)	ND(10)	ND(10)	2.9	ND(10)	ND(10)	ND(10)	**4.2**	ND(10)	ND(10)
Napthalene	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	**39.0**	21	ND(10)
4-Chloroaniline	ND(20)	R	ND(20)	ND(20)	ND(20)	ND(20)	ND(20)	ND(20)	**24**	7.3	2.8
Pentachlorophenol	NR	ND(50)	ND(50)	ND(50)	NR	**6.4**	5.9	ND(50)	NR	ND(50)	NR
Phenol	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	**14.0**	3.4	2.8
Total SVOCs					20.2	6.4	5.9		159.2	87.5	30.7
Total VOCs and SVOCs					21.2	12.2	18	5	**5,249**	4,748	2,414

Notes:

1) Data Source: Sauget Area 2 RI/FS Data Report
2) Closest Vertical Groundwater Profile: AA-P1
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: July 2002
6) Selected Screen Depth: 104 ft bgs

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 12
Table 2.11

	Sample Depth				
VOCs, ug/l	40 (ft bgs)	60 (ft bgs)	80 (ft bgs)	100 (ft bgs)	120 (ft bgs)
Benzene	ND(1)	0.62	ND(50)	18	**23**
Carbon Disulfide	ND(1)	0.34	ND(50)	**750**	10
Chlorobenzene	ND(1)	78	5,100	**8,200**	6,700
Chloromethane	ND(1)	0.3	21	ND(25)	ND(25)
Total VOCs		79.26	5,121	**8,968**	6,733
SVOCs, ug/l					
1,2-Dichlorobenzene	ND(10)	ND(10)	7.9	8.6	**15**
1,3-Dichlorobenzene	ND(10)	ND(10)	ND(10)	6.8	**12**
1,4-Dichlorobenzene	ND(10)	ND(10)	120	180	**360**
2,4-Dichlorophenol	ND(10)	ND(10)	1.8	2.3	**4**
2,4-Dimethylphenol	ND(10)	ND(10)	ND(10)	7.7	ND(10)
2-Chlorophenol	ND(10)	2.3	49	56	42
Dibenzo(a,h)anthracene	ND(10)	ND(10)	ND(10)	2.2	ND(10)
Total SVOCs		2.3	178.7	263.6	**433**
Total VOCs and SVOCs		81.56	5,299.70	**9,231.60**	7,166.00

Notes:

1) Data Source: W.G. Krummrich Facility CMS Data Report
2) Closest Vertical Groundwater Profile: AA-GWM-S2
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: July 2002
6) Selected Screen Depth: 100 ft bgs

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 13
Table 2.12

	Sample Depth										
VOCs, ug/l	24 (ft bgs)	34 (ft bgs)	44 (ft bgs)	54 (ft bgs)	64 (ft bgs)	74 (ft bgs)	84 (ft bgs)	94 (ft bgs)	104 (ft bgs)	114 (ft bgs)	124 (ft bgs)
1,2-Dichloroethene	0.3	ND(2)	ND(2)	ND(2)	ND(1)	0.53	0.84	0.74	**1.5**	0.57	ND(2)
2-Butanone	ND(10)	ND(10)	ND(10)	11	ND(10)	ND(10)	**8.4**	ND(10)	ND(10)	ND(10)	ND(15)
Benzene	**3**	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)
Chlorobenzene	ND(1)	1.7	ND(1)	0.44	ND(1)	4.8	22	27	70	**410**	78
Chloroethane	ND(1)	**3.3**	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)
Total VOCs	3.3	5.0		11.44		5.33	31.2	27.74	71.5	**410.57**	78
SVOCs, ug/l											
1,2-Dichlorobenzene	**1.5**	1.4	ND(20)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)
2-Chlorophenol	ND(10)	ND(10)	ND(20)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	**7.1**	ND(10)
Bis(2-chloroethyl)ether	**1.8**	1.4	ND(20)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)
Bis(2-ethylhexyl)phthalate	ND(10)	ND(10)	ND(20)	**3.5**	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)
Napthalene	ND(10)	ND(10)	ND(20)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	**3.3**	ND(10)
Total SVOCs	3.3	2.8		3.5					0.0	**10.4**	
Total VOCs and SVOCs	6.6	7.8		14.94		5.33	31.2	27.74	71.5	**420.97**	78

Notes:

1) Data Source: Sauget Area 2 RI/FS Data Report
2) Closest Vertical Groundwater Profile: AA-S1
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: July 2002
6) Selected Screen Depth: 114 ft bgs

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selections
Monitoring Wells 14 & 15
Table 2.13

	Sample Depth										
VOCs, ug/l	32 (ft bgs)	42 (ft bgs)	52 (ft bgs)	62 (ft bgs)	72 (ft bgs)	82 (ft bgs)	92 (ft bgs)	102 (ft bgs)	112 (ft bgs)	122 (ft bgs)	126 (ft bgs)
4-Methyl-2-pentanone	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	1.3	ND(10)
Benzene	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	0.54	ND(1)	1.3	ND(1)	ND(1)	ND(1)
Chlorobenzene	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	ND(1)	0.45	6.1	ND(1)	ND(1)
Total VOCs						0.54		1.75	**6.1**	1.30	
SVOCs, ug/l											
NONE DETECTED											
Total SVOCs											
Total VOCs and SVOCs											

Notes:

1) Data Source: Sauget Area 2 RI/FS Data Report
2) Closest Vertical Groundwater Profile: AA-P3
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: August 2002
6) Selected Screen Depth: 112 ft bgs

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 16
Table 2.14

	Sample Depth					
VOCs, ug/l	40 (ft bgs)	60 (ft bgs)	80 (ft bgs)	100 (ft bgs)	120 (ft bgs)	140 (ft bgs)
1,1-Dichloroethane	ND(1)	ND(1)	ND(5)	ND(25)	**14**	13
Benzene	0.59	ND(1)	**79**	76	68	56
Chlorobenzene	2.7	ND(1)	620	2,800	**3,700**	3,000
Chloroethane	ND(1)	ND(1)	2.9	**11**	ND(25)	ND(25)
Toluene	ND(1)	ND(1)	**3.6**	ND(25)	ND(25)	ND(25)
Trichloroethene	ND(1)	ND(1)	ND(5)	ND(25)	ND(25)	**15**
Vinyl Chloride	ND(1)	ND(1)	ND(5)	ND(25)	**27**	**27**
Total VOCs	3.29		705.5	2,887	**3,809**	3,111
SVOCs, ug/l						
1,2-Dichlorobenzene	ND(10)	ND(10)	ND(10)	3.3	**13**	7.3
1,3-Dichlorobenzene	ND(10)	ND(10)	ND(10)	ND(10)	**5.5**	2.7
1,4-Dichlorobenzene	ND(10)	ND(10)	ND(10)	17	**180**	93
2,4-Dichlorophenol	ND(10)	ND(10)	ND(10)	ND(10)	**5.3**	2.3
2-Chlorophenol	ND(10)	ND(10)	8.5	40	**46**	36
4-Chloroaniline	ND(20)	ND(20)	2.2	**12**	6.3	5
Naphthalene	ND(10)	ND(10)	**3.8**	ND(10)	ND(10)	ND(10)
Total SVOCs			16.7	84.3	**262.4**	151.3
Total VOCs and SVOCs			722.2	2,971.3	**4,071.4**	3,262.3

Notes:

1) Data Source: W.G. Krummrich CMS Data Report
2) Closest Vertical Groundwater Profile: AA-GWM-S1
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: July 2002
6) Selected Screen Depth: 120 ft bgs

W.G. Krummrich Facility, Sauget, Illinois
Plume Stability Monitoring Plan
Screen Depth Selection
Monitoring Well 17
Table 2.15

	Sample Depth								
VOCs, ug/l	50 (ft bgs)	60 (ft bgs)	70 (ft bgs)	80 (ft bgs)	90 (ft bgs)	100 (ft bgs)	110 (ft bgs)	120 (ft bgs)	127 (ft bgs)
1,2-Dichloroethane	**5.6**	4.9	1.1	2.2	2.9	ND(1)	ND(1)	ND(2)	ND(5)
Benzene	2.4	ND(1)	ND(1)	ND(2)	1.4	ND(1)	10	28	**33**
Chlorobenzene	130	240	100	130	220	200	130	310	**1,100**
Ethylbenzene	1	ND(1)	ND(1)	ND(2)	ND(1)	ND(1)	0.89	2.1	**6.9**
Toluene	ND(1)	ND(1)	ND(1)	ND(2)	ND(1)	ND(1)	0.39	ND(2)	**1.5**
Vinyl Chloride	0.72	ND(1)	ND(1)	ND(2)	ND(1)	ND(1)	3.2	2.9	**4.6**
Total VOCs	139.72	244.9	101.1	132.2	224.3	200	144.48	343.0	**1,146**
SVOCs, ug/l									
1,2-Dichlorobenzene	11	8	4	5.6	ND(10)	ND(10)	3	7.4	**440**
1,3-Dichlorobenzene	**2.5**	1.5	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	**2.5**
1,4-Dichlorobenzene	**30**	21	9.6	12	12	7.8	3.2	6.1	22
2,4-Dichlorophenol	1	ND(10)	ND(10)	1	ND(10)	ND(10)	ND(10)	ND(10)	**12**
2-Chlorophenol	3.2	3	1.6	2.4	3.7	4.6	3.6	**10**	5.6
4-Chloroaniline	130	80	14	55	110	ND(10)	ND(10)	36	**3,500**
Benzo(b)fluoranthene	ND(10)	ND(10)	ND(10)	**3.7**	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)
Benzo(g,h,i)perylene	ND(10)	ND(10)	ND(10)	**1.2**	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)
Dibenzo(a,h)anthracene	ND(10)	ND(10)	ND(10)	**2.8**	ND(10)	ND(10)	ND(10)	1	ND(10)
Indeno(1,2,3-cd)pyrene	ND(10)	ND(10)	ND(10)	0.88	ND(10)	ND(10)	ND(10)	**1**	ND(10)
Napthalene	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	2	**5.9**	3.1
Phenol	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	ND(10)	**3.4**	2
Total SVOCs	177.7	113.5	29.2	84.6	125.7	12.4	11.8	70.8	**3,987**
Total VOCs and SVOCs	317.42	358.4	130.3	216.8	350.0	212.4	156.28	413.8	**5,133**

Notes:

1) Data Source: Sauget Area 2 RI/FS Data Report
2) Closest Vertical Groundwater Profile: AA-Q1
3) Highest Detected Concentrations in Bold
4) Concentrations < 1 ug/l Not Reported
5) Sample Collection Date: July 2002
6) Selected Screen Depth: 127 ft bgs

TABLE 3.1
GROUNDWATER ELEVATION PIEZOMETER INFORMATION

Piezometer ID	Screened Hydrostratigraphic Unit	Top of Piezometer Elevation (ft MSL)	Total Piezometer Depth (ft btoc)	Bottom of Piezometer Elevation (ft MSL)	Screened Interval Depth (ft btoc)			Screened Interval Elevation (ft MSL)		
GWE-1S	SHU	416.54	25.71	390.83	15.71	-	25.71	400.83	-	390.83
GWE-1M	DHU	416.26	79.43	336.83	69.43	-	79.43	346.83	-	336.83
GWE-1D	DHU	416.39	129.56	286.83	119.56	-	129.56	296.83	-	286.83
GWE-2S	SHU	417.48	26.66	390.82	16.66	-	26.66	400.82	-	390.82
GWE-2M	DHU	417.57	77.75	339.82	67.75	-	77.75	349.82	-	339.82
GWE-2D	DHU	417.56	136.74	280.82	126.74	-	136.74	290.82	-	280.82
GWE-3S	SHU	417.8	37.77	380.03	27.77	-	37.77	390.03	-	380.03
GWE-3M	DHU	417.84	78.31	339.53	68.31	-	78.31	349.53	-	339.53
GWE-3D	DHU	417.66	114.63	303.03	104.63	-	114.63	313.03	-	303.03
GWE-4S	MHU	406.06	30.59	375.47	20.59	-	30.59	385.47	-	375.47
GWE-4M	MHU	405.86	49.76	356.10	43.76	-	49.76	362.10	-	356.10
GWE-4D	DHU	405.92	78.11	327.81	72.11	-	78.11	333.81	-	327.81
GWE-5S	SHU	408.76	28.75	380.01	18.75	-	28.75	390.01	-	380.01
GWE-5M	MHU	408.94	48.41	360.53	42.41	-	48.41	366.53	-	360.53
GWE-5D	DHU	409.05	78.93	330.12	72.93	-	78.93	336.12	-	330.12
GWE-6S	MHU	415.25	35.52	379.73	25.52	-	35.52	389.73	-	379.73
GWE-6M	MHU	415.27	56.26	359.01	50.26	-	56.26	365.01	-	359.01
GWE-6D	DHU	415.25	98.18	317.07	92.18	-	98.18	323.07	-	317.07
GWE-7S	SHU	411.25	30.60	380.65	20.60	-	30.60	390.65	-	380.65
GWE-7M	DHU	411.16	66.51	344.65	60.51	-	66.51	350.65	-	344.65
GWE-7D	DHU	411.37	81.78	329.59	75.78	-	81.78	335.59	-	329.59
GWE-8S	MHU	421.86	52.78	369.08	42.78	-	52.78	379.08	-	369.08
GWE-8M	DHU	422.02	93.94	328.08	83.94	-	93.94	338.08	-	328.08
GWE-8D	DHU	422	131.92	290.08	121.92	-	131.92	300.08	-	290.08
GWE-9S	SHU	408.62	25.88	382.74	15.88	-	25.88	392.74	-	382.74
GWE-9M	DHU	408.49	69.75	338.74	59.75	-	69.75	348.74	-	338.74
GWE-9D	DHU	408.61	108.87	299.74	98.87	-	108.87	309.74	-	299.74
GWE-10S	SHU	413.76	29.79	383.97	19.79	-	29.79	393.97	-	383.97
GWE-10M	DHU	413.62	74.65	338.97	64.65	-	74.65	348.97	-	338.97
GWE-10D	DHU	413.7	115.23	298.47	105.23	-	115.23	308.47	-	298.47
GWE-14S	MHU	423.06	47.74	375.32	37.74	-	47.74	385.32	-	375.32
GWE-14M	MHU	423.09	51.11	371.98	45.11	-	51.11	377.98	-	371.98
GWE-14D	DHU	423.15	82.85	340.30	76.85	-	82.85	346.30	-	340.30
GWE-15S	MHU	408.76	38.07	370.69	32.07	-	38.07	376.69	-	370.69
GWE-15M	MHU	409.22	47.41	361.81	41.41	-	47.41	367.81	-	361.81
GWE-15D	DHU	409.08	81.89	327.19	75.89	-	81.89	333.19	-	327.19
GWE-22S	SHU	417.02	27.60	389.42	17.60	-	27.60	399.42	-	389.42
GWE-22M	DHU	417.10	75.18	341.92	65.18	-	75.18	351.92	-	341.92
GWE-22D	DHU	417.02	117.60	299.42	107.60	-	117.60	309.42	-	299.42
GWE-23S	SHU	403.82	21.85	381.97	11.85	-	21.85	391.97	-	381.97
GWE-23M	DHU	403.84	68.87	334.97	58.87	-	68.87	344.97	-	334.97
GWE-23D	DHU	403.81	110.84	292.97	100.84	-	110.84	302.97	-	292.97
GWE-24S	SHU	402.75	18.75	384.00	8.75	-	18.75	394.00	-	384.00
GWE-24M	DHU	402.82	64.32	338.50	54.32	-	64.32	348.50	-	338.50
GWE-24D	DHU	402.71	104.71	298.00	94.71	-	104.71	308.00	-	298.00

Notes:

1) MSL = Mean Sea Level

2) btoc = below top of casing

3) Piezometer construction information is not currently available for GWE-11, GWE-12, GWE-13, GWE-16, GWE-17, GWE-18, GWE-19, GWE-20, and GWE-21.

4) The Screened Hydrostratigraphic Unit is based on the Bottom of Piezometer Evaluation (BPE). SHU indicates a BPE between 395 to 380 feet MSL. MHU indicates a BPE between 380 to 350 feet MSL. DHU indicates a BPE less than 350 feet MSL.

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Metals							
Aluminum	7429-90-5	6010 (3010)	30	200.00	3500.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Antimony	7440-36-0	6010 (3010)	5.1	20.00	6.00	IEPA TACO Class I	No
Arsenic	7440-38-2	6010 (3010)	5.2	10.00	50.00	IEPA TACO Class I	Yes
Barium	7440-39-3	6010 (3010)	1.2	10.00	2000.00	IEPA TACO Class I	Yes
Beryllium	7440-41-7	6010 (3010)	0.14	4.00	4.00	IEPA TACO Class I	Yes
Boron	7440-42-8	6010 (3010)	17	50.00	2000.00	IEPA TACO Class I	Yes
Cadmium	7440-43-9	6010 (3010)	1.0	5.00	5.00	IEPA TACO Class I	Yes
Calcium	7440-70-2	6010 (3010)	50	500.00	NA	Not Available	NA
Chromium	7440-47-3	6010 (3010)	0.95	10.00	100.00	IEPA TACO Class I	Yes
Cobalt	7440-48-4	6010 (3010)	1.3	10.00	1000.00	IEPA TACO Class I	Yes
Copper	7440-50-8	6010 (3010)	1.7	20.00	650.00	IEPA TACO Class I	Yes
Iron	15438-31-0	6010 (3010)	28	50.00	5000.00	IEPA TACO Class I	Yes
Lead	7439-92-1	6010 (3010)	3.5	5.00	7.50	IEPA TACO Class I	Yes
Magnesium	7439-95-4	6010 (3010)	9.8	500.00	NA	Not Available	NA
Manganese	7439-96-5	6010 (3010)	2.0	10.00	150.00	IEPA TACO Class I	Yes
Mercury (CVAA)	7439-97-6	7470	0.080	0.20	2.00	IEPA TACO Class I	Yes
Molybdenum	7439-98-7	6010 (3010)	3.6	10.00	NA	Not Available	NA
Nickel	7440-02-0	6010 (3010)	1.5	40.00	100.00	IEPA TACO Class I	Yes
Potassium	7440-09-7	6010 (3010)	15	1000.00	NA	Not Available	NA
Selenium	7782-49-2	6010 (3010)	5.4	10.00	50.00	IEPA TACO Class I	Yes
Silver	7440-22-4	6010 (3010)	1.0	10.00	50.00	IEPA TACO Class I	Yes
Sodium	7440-23-5	6010 (3010)	300	1000.00	NA	Not Available	NA
Strontium	7440-24-6	6010 (3010)	1.5	10.00	4200.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Thallium	7440-28-0	6010 (3010)	11	25.00	2.00	IEPA TACO Class I	No

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Tin	7440-31-5	6010 (3010)	6.3	50.00	4200.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Titanium	7440-32-6	6010 (3010)	0.32	10.00	NA	Not Available	NA
Vanadium	7440-62-2	6010 (3010)	1.4	10.00	49.00	IEPA TACO Class I	Yes
Zinc	7440-66-6	6010 (3010)	3.0	20.00	5000.00	IEPA TACO Class I	Yes
PCB Homologs by GC/MS (SIM)							
Monochlorobiphenyls	Not Available	680	0.010	0.10	NA	Not Available	NA
Dichlorobiphenyls	2050-68-2	680	0.012	0.10	NA	Not Available	NA
Trichlorobiphenyls	Not Available	680	0.012	0.10	NA	Not Available	NA
Tetrachlorobiphenyls	26914-33-0	680	0.023	0.20	NA	Not Available	NA
Pentachlorobiphenyls	25429-29-2	680	0.026	0.20	NA	Not Available	NA
Hexachlorobiphenyls	26601-64-9	680	0.024	0.20	NA	Not Available	NA
Heptachlorobiphenyls	28655-71-2	680	0.037	0.30	NA	Not Available	NA
Octachlorobiphenyls	74472-52-9 & 55722-26-4	680	0.036	0.30	NA	Not Available	NA
Nonachlorobiphenyls	53742-07-7	680	0.062	0.50	NA	Not Available	NA
Decachlorobiphenyl	2051-24-3	680	0.084	0.50	NA	Not Available	NA
Surrogate							
Decachlorobiphenyl-13C12	2051-24-3	680	NA	NA	NA	Not Available	NA
Chlorinated Pesticides In Groundwater by GC/EC							
4,4'-DDD	72-54-8	8081 (3520)	0.011	0.10	14.00	IEPA TACO Class I	Yes
4,4'-DDE	72-55-9	8081 (3520)	0.012	0.10	10.00	IEPA TACO Class I	Yes
4,4'-DDT (MS)	50-29-3	8081 (3520)	0.014	0.10	6.00	IEPA TACO Class I	Yes
Aldrin (MS)	309-00-2	8081 (3520)	0.0053	0.05	14.00	IEPA TACO Class I	Yes
alpha BHC	319-84-6	8081 (3520)	0.0086	0.05	1.10	IEPA TACO Class I	Yes
alpha Chlordane	5103-71-9	8081 (3520)	0.0068	0.05	NA	Not Available	NA
beta BHC	319-85-7	8081 (3520)	0.0057	0.05	NA	Not Available	NA
Chlorobenzilate	510-15-6	8081 (3520)	0.19	0.50	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
delta BHC	319-86-8	8081 (3520)	0.0098	0.05	NA	Not Available	NA
Dieldrin (MS)	60-57-1	8081 (3520)	0.010	0.10	9.00	IEPA TACO Class I	Yes
Endosulfan I	959-98-8	8081 (3520)	0.0064	0.05	42.00	IEPA TACO Class I	Yes
Endosulfan II	33213-65-9	8081 (3520)	0.011	0.10	42.00	IEPA TACO Class I	Yes
Endosulfan sulfate	1031-07-8	8081 (3520)	0.010	0.10	42.00	IEPA TACO Class I	Yes
Endrin (MS)	72-20-8	8081 (3520)	0.010	0.10	2.00	IEPA TACO Class I	Yes
Endrin aldehyde	7421-93-4	8081 (3520)	0.017	0.10	NA	Not Available	NA
Endrin ketone	53494-70-5	8081 (3520)	0.016	0.10	NA	Not Available	NA
gamma BHC (Lindane) (MS)	58-89-9	8081 (3520)	0.0055	0.05	0.20	IEPA TACO Class I	Yes
gamma Chlordane	12789-03-6	8081 (3520)	0.013	0.05	NA	Not Available	NA
Heptachlor (MS)	76-44-8	8081 (3520)	0.0076	0.05	0.40	IEPA TACO Class I	Yes
Heptachlor epoxide	1024-57-3	8081 (3520)	0.015	0.05	0.20	IEPA TACO Class I	Yes
Isodrin	465-73-6	8081 (3520)	0.0082	0.05	NA	Not Available	NA
Kepone	143-50-0	8081 (3520)	0.12	1.00	NA	Not Available	NA
Methoxychlor	72-43-5	8081 (3520)	0.024	0.50	40.00	IEPA TACO Class I	Yes
Technical Chlordane	57-74-9	8081 (3520)	0.17	0.50	2.00	IEPA TACO Class I	Yes
Toxaphene	8001-35-2	8081 (3520)	0.88	5.00	3.00	IEPA TACO Class I	No
Surrogates							
Decachlorobiphenyl	2051-24-3	8081 (3520)	NA	NA	NA	Not Available	NA
2,4,5,6-Tetrachloro-m-xylene	877-09-8	8081 (3520)	NA	NA	NA	Not Available	NA
Non-routine Compounds							
2,4'-DDD	72-54-8	8081 (3520)	0.073	0.10	14.00	IEPA TACO Class I	Yes
2,4'-DDE	72-55-9	8081 (3520)	0.068	0.10	10.00	IEPA TACO Class I	Yes
2,4'-DDT	50-29-3	8081 (3520)	0.071	0.10	6.00	IEPA TACO Class I	Yes
Chlorinated Herbicides by GC/EC							
2,4,5-T (MS)	93-76-5	8151	0.080	0.50	280.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
2,4,5-TP (Silvex) (MS)	93-72-1	8151	0.033	0.50	50.00	IEPA TACO Class I	Yes
2,4-D (MS)	94-75-7	8151	0.11	0.50	70.00	IEPA TACO Class I	Yes

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
2,4-DB	94-82-6	8151	0.057	0.50	NA	Not Available	NA
Ammonia (as N)	7664-41-7	350.1 / 4500-NH3-H	0.015	0.03	NA	Not Available	NA
Dalapon	75-99-0	8151	0.24	10.00	200.00	IEPA TACO Class I	Yes
Dicamba	1918-00-9	8151	0.048	0.50	210.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Dichlorprop	120-36-5	8151	0.030	0.50	NA	Not Available	NA
Dinoseb	88-85-7	8151	0.40	6.00	7.00	IEPA TACO Class I	Yes
MCPA	94-74-6	8151	12	120.00	3.50	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
MCPP	93-65-2	8151	10	120.00	7.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Pentachlorophenol	87-86-5	8151	0.18	0.25	1.00	IEPA TACO Class I	Yes
Surrogate							
2,4-Dichlorophenylacetic acid (DCAA)	19719-28-9	8151	NA	NA	NA	Not Available	NA
Non-routine Compounds							
Picloram	1918-02-1	8151	0.089	0.50	500.00	IEPA TACO Class I	Yes
Volatiles in Groundwater by GC/MS							
1,1,1,2-Tetrachloroethane	630-20-6	8260 (5030)	0.53	1.00	210.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
1,1,1-Trichloroethane	71-55-6	8260 (5030)	0.79	1.00	200.00	IEPA TACO Class I	Yes
1,1,2,2-Tetrachloroethane	79-34-5	8260 (5030)	0.21	1.00	420.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	8260 (5030)	0.10	1.00	NA	Not Available	NA
1,1,2-Trichloroethane	79-00-5	8260 (5030)	0.37	1.00	5.00	IEPA TACO Class I	Yes

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
1,1-Dichloroethane	75-34-3	8260 (5030)	0.56	1.00	700.00	IEPA TACO Class I	Yes
1,1-Dichloroethene (MS)	75-35-4	8260 (5030)	0.93	1.00	7.00	IEPA TACO Class I	Yes
1,1-Dichloropropene	563-58-6	8260 (5030)	0.80	1.00	NA	Not Available	NA
1,2,3-Trichlorobenzene	87-61-6	8260 (5030)	0.35	1.00	70.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
1,2,3-Trichloropropane	96-18-4	8260 (5030)	0.44	1.00	1.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
1,2,4-Trichlorobenzene	120-82-1	8260 (5030)	0.28	1.00	70.00	IEPA TACO Class I	Yes
1,2,4-Trimethylbenzene	95-63-6	8260 (5030)	0.44	1.00	350.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
1,2-Dibromo-3-chloropropane	96-12-8	8260 (5030)	0.65	1.00	0.20	IEPA TACO Class I	No
1,2-Dibromoethane	106-93-4	8260 (5030)	0.34	1.00	0.05	IEPA TACO Class I	No
1,2-Dichlorobenzene	95-50-1	8260 (5030)	0.21	1.00	600.00	IEPA TACO Class I	Yes
1,2-Dichloroethane	107-06-2	8260 (5030)	0.28	1.00	5.00	IEPA TACO Class I	Yes
1,2-Dichloroethenes, Total (sum of cis- and trans- isomers)	540-59-0	8260 (5030)	1.2	2.00	55.00	Region 3 (Tap Water)	Yes
1,2-Dichloropropane	78-87-5	8260 (5030)	0.26	1.00	5.00	IEPA TACO Class I	Yes
1,3,5-Trimethylbenzene	108-67-8	8260 (5030)	0.49	1.00	350.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
1,3-Dichlorobenzene	541-73-1	8260 (5030)	0.28	1.00	5.50	Region 9 (Tap Water)	Yes
1,3-Dichloropropane	142-28-9	8260 (5030)	0.31	1.00	NA	Not Available	NA
1,4-Dichlorobenzene	106-46-7	8260 (5030)	0.44	1.00	75.00	IEPA TACO Class I	Yes
2,2-Dichloropropane	594-20-7	8260 (5030)	0.86	1.00	NA	Not Available	NA
2-Butanone (methyl ethyl ketone-MEK)	78-93-3	8260 (5030)	0.72	10.00	4.20	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
2-Chloro-1,3-butadiene (Chloroprene)	126-99-8	8260 (5030)	0.40	1.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
2-Chloroethyl vinyl ether	110-75-8	8260 (5030)	0.55	10.00	NA	Not Available	NA
2-Chlorotoluene	95-49-8	8260 (5030)	0.58	1.00	140.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
2-Hexanone	591-78-6	8260 (5030)	0.39	10.00	0.28	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
3-Chloropropene (Allyl chloride)	107-05-1	8260 (5030)	0.35	1.00	NA	Not Available	NA
4-Chlorotoluene	106-43-4	8260 (5030)	0.44	1.00	NA	Not Available	NA
4-Methyl-2-pentanone (MIBK)	108-10-1	8260 (5030)	0.45	10.00	560.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	Yes
Acetone	67-64-1	8260 (5030)	7.3	25.00	700.00	IEPA TACO Class I	Yes
Acetonitrile	75-05-8	8260 (5030)	0.23	40.00	NA	Not Available	NA
Acrolein	107-02-8	8260 (5030)	12	20.00	3.50	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Acrylonitrile	107-13-1	8260 (5030)	3.2	20.00	5.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Benzene (MS)	71-43-2	8260 (5030)	0.54	1.00	5.00	IEPA TACO Class I	Yes
Bromobenzene	108-86-1	8260 (5030)	0.45	1.00	NA	Not Available	NA
Bromochloromethane	74-97-5	8260 (5030)	0.25	1.00	NA	Not Available	NA
Bromodichloromethane	75-27-4	8260 (5030)	0.42	1.00	0.20	IEPA TACO Class I	No
Bromoform	75-25-2	8260 (5030)	0.36	1.00	1.00	IEPA TACO Class I	Yes
Bromomethane	74-83-9	8260 (5030)	0.93	1.00	50.00	IEPA TACO Class I	Yes
Carbon disulfide	75-15-0	8260 (5030)	0.75	1.00	700.00	IEPA TACO Class I	Yes
Carbon tetrachloride	56-23-5	8260 (5030)	0.91	1.00	5.00	IEPA TACO Class I	Yes
Chlorobenzene (MS)	108-90-7	8260 (5030)	0.41	1.00	100.00	IEPA TACO Class I	Yes

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Chloroethane	75-00-3	8260 (5030)	0.89	1.00	2.80	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Chloroform	67-66-3	8260 (5030)	0.52	1.00	0.20	IEPA TACO Class I	No
Chloromethane	74-87-3	8260 (5030)	0.53	1.00	0.03	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
cis-1,2-Dichloroethene	156-59-2	8260 (5030)	0.55	1.00	70.00	IEPA TACO Class I	Yes
cis-1,3-Dichloropropene	10061-01-5	8260 (5030)	0.25	1.00	0.00	None Found	No
Dibromochloromethane	124-48-1	8260 (5030)	0.40	1.00	140.00	IEPA TACO Class I	Yes
Dibromomethane	74-95-3	8260 (5030)	0.33	1.00	70.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Dichlorodifluoromethane	75-71-8	8260 (5030)	0.73	1.00	1400.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Ethyl methacrylate	97-63-2	8260 (5030)	0.33	1.00	NA	Not Available	NA
Ethylbenzene	100-41-4	8260 (5030)	0.62	1.00	700.00	IEPA TACO Class I	Yes
Hexachlorobutadiene	87-68-3	8260 (5030)	0.50	1.00	NA	Not Available	NA
Iodomethane	74-88-4	8260 (5030)	0.96	5.00	1050.00	Solutia Queeny	Yes
Isobutyl alcohol	78-83-1	8260 (5030)	13	40.00	2100.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Isopropylbenzene	98-82-8	8260 (5030)	0.66	1.00	700.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
m&p-Xylene	108-38-3, 106-42-3	8260 (5030)	1.3	2.00	10000.00	IEPA TACO Class I - Surrogate Chemical: Xylenes, Total	Yes
Methacrylonitrile	126-98-7	8260 (5030)	9.1	20.00	NA	Not Available	NA
Methyl t-butyl ether (MTBE)	1634-04-4	8260 (5030)	0.45	10.00	70.00	IEPA TACO Class I	Yes
Methylene chloride	75-09-2	8260 (5030)	0.44	5.00	5.00	IEPA TACO Class I	Yes
Methylmethacrylate	80-62-6	8260 (5030)	0.36	1.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I. Screening Value (ug/L)	Ref Description	Screening Value > RL?
Naphthalene	91-20-3	8260 (5030)	0.12	5.00	140.00	IEPA TACO Class I	Yes
n-Butylbenzene	104-51-8	8260 (5030)	0.38	1.00	70.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
n-Propylbenzene	103-65-1	8260 (5030)	0.45	1.00	70.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
o-Xylene	95-47-6	8260 (5030)	0.49	1.00	10000.00	IEPA TACO Class I - Surrogate Chemical: Xylenes, Total	Yes
Pentachloroethane	76-01-7	8260 (5030)	1.0	5.00	NA	Not Available	NA
p-Isopropyltoluene	99-87-6	8260 (5030)	0.52	1.00	NA	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Propionitrile (ethylcyanide)	107-12-0	8260 (5030)	10	20.00	NA	Not Available	NA
sec-Butylbenzene	135-98-8	8260 (5030)	0.60	1.00	NA	Not Available	NA
Styrene	100-42-5	8260 (5030)	0.42	1.00	100.00	IEPA TACO Class I	Yes
tert-Butylbenzene	98-06-6	8260 (5030)	0.63	1.00	70.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Tetrachloroethene	127-18-4	8260 (5030)	0.75	1.00	5.00	IEPA TACO Class I	Yes
Toluene (MS)	108-88-3	8260 (5030)	0.62	1.00	1000.00	IEPA TACO Class I	Yes
trans-1,2-Dichloroethene	156-60-5	8260 (5030)	0.80	1.00	100.00	IEPA TACO Class I	Yes
trans-1,3-Dichloropropene	10061-02-6	8260 (5030)	0.36	1.00	NA	Not Available	NA
trans-1,4-Dichloro-2-butene	110-57-6	8260 (5030)	0.80	2.00	NA	Not Available	NA
Trichloroethene(MS)	79-01-6	8260 (5030)	0.71	1.00	5.00	IEPA TACO Class I	Yes
Trichlorofluoromethane	75-69-4	8260 (5030)	0.96	1.00	2100.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Vinyl acetate	108-05-4	8260 (5030)	0.70	2.00	7000.00	IEPA TACO Class I	Yes
Vinyl chloride	75-01-4	8260 (5030)	0.92	1.00	2.00	IEPA TACO Class I	Yes
Xylenes (total)	1330-20-7	8260 (5030)	1.6	2.00	10000.00	IEPA TACO Class I	Yes

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Surrogates							
p-Bromofluorobenzene	460-00-4	8260 (5030)	NA	NA	NA	Not Available	NA
Dibromofluoromethane	1868-53-7	8260 (5030)	NA	NA	NA	Not Available	NA
Toluene-d8	108-88-3	8260 (5030)	NA	NA	1000.00	IEPA TACO Class I	No
Non-Routine Analytes							
Cyclohexane	110-82-7	8260 (5030)	0.25	1.00	NA	Not Available	NA
1-Chlorohexane	544-10-5	8260 (5030)	0.42	1.00	NA	Not Available	NA
Cyclohexanone	108-94-1	8260 (5030)	6.9	50.00	35000.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Diethyl ether		8260 (5030)	0.14	2.00	NA	Not Available	NA
Furan	110-00-9	8260 (5030)	0.14	10.00	NA	Not Available	NA
Methyl acetate	79-20-9	8260 (5030)	0.80	10.00	7000.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Methyl cyclohexane	108-87-2	8260 (5030)	0.22	10.00	NA	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Tetrahydrofuran	109-99-9	8260 (5030)	0.56	10.00	1400.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Semivolatiles in Groundwater by GC/MS							
1,2,4,5-Tetrachlorobenzene	95-94-3	8270 (3520)	1.2	10.00	NA	Not Available	NA
1,2,4-Trichlorobenzene (MS)	120-82-1	8270 (3520)	0.54	10.00	70.00	IEPA TACO Class I	Yes
1,2-Dichlorobenzene	95-50-1	8270 (3520)	1.0	10.00	600.00	IEPA TACO Class I	Yes
1,2-Diphenyl hydrazine	122-66-7	8270 (3520)	0.97	10.00	NA	Not Available	NA
1,3,5-Trinitrobenzene	99-35-4	8270 (3520)	0.62	10.00	210.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	Yes
1,3-Dichlorobenzene	541-73-1	8270 (3520)	0.55	10.00	5.50	Region 9 (Tap Water)	No

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
1,4-Dichlorobenzene(MS)	106-46-7	8270 (3520)	0.52	10.00	75.00	IEPA TACO Class I	Yes
1,4-Dioxane	123-91-1	8270 (3520)	2.3	10.00	1.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	No
1,4-Naphthoquinone	130-15-4	8270 (3520)	0.67	10.00	NA	Not Available	NA
1-Methylnaphthalene	90-12-0	8270 (3520)	0.62	10.00	28.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	Yes
1-Naphthylamine	134-32-7	8270 (3520)	1.2	10.00	NA	Not Available	NA
2,3,4,6-Tetrachlorophenol	58-90-2	8270 (3520)	0.47	10.00	NA	Not Available	NA
2,4,5-Trichlorophenol	95-95-4	8270 (3520)	0.63	10.00	700.00	IEPA TACO Class I	Yes
2,4,6-Trichlorophenol	88-06-2	8270 (3520)	0.70	10.00	10.00	IEPA TACO Class I	Yes
2,4-Dichlorophenol	120-83-2	8270 (3520)	0.66	10.00	21.00	IEPA TACO Class I	Yes
2,4-Dimethylphenol	105-67-9	8270 (3520)	1.0	10.00	140.00	IEPA TACO Class I	Yes
2,4-Dinitrophenol	51-28-5	8270 (3520)	5.0	50.00	14.00	IEPA TACO Class I	No
2,4-Dinitrotoluene (MS)	121-14-2	8270 (3520)	0.56	10.00	0.02	IEPA TACO Class I	No
2,6-Dichlorophenol	87-65-0	8270 (3520)	0.69	10.00	NA	Not Available	NA
2,6-Dinitrotoluene	606-20-2	8270 (3520)	0.57	10.00	0.31	IEPA TACO Class I	No
2-Acetylaminofluorene	53-96-3	8270 (3520)	1.4	10.00	NA	Not Available	NA
2-Chloronaphthalene	91-58-7	8270 (3520)	0.62	10.00	NA	Not Available	NA
2-Chlorophenol (MS)	95-57-8	8270 (3520)	0.72	10.00	35.00	IEPA TACO Class I	Yes
2-Methyl phenol	95-48-7	8270 (3520)	0.70	10.00	350.00	IEPA TACO Class I	Yes
2-Methylnaphthalene	91-57-6	8270 (3520)	0.53	10.00	140.00	IEPA TACO Class I - Surrogate Chemical: Napthalene (per USEPA National Center for Environmental Assessment)	Yes
2-Naphthylamine	91-59-8	8270 (3520)	0.64	10.00	NA	Not Available	NA
2-Nitroaniline	88-74-4	8270 (3520)	0.69	50.00	1.00	Region 9 (Tap Water)	No
2-Nitrophenol	88-75-5	8270 (3520)	0.73	10.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
2-Picoline	109-06-8	8270 (3520)	0.87	10.00	7.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	No
3- and 4-Methyl phenol	106-44-5	8270 (3520)	0.66	10.00	35.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	Yes
3,3'-Dichlorobenzidine	91-94-1	8270 (3520)	1.0	20.00	20.00	IEPA TACO Class I	Yes
3,3'-Dimethylbenzidine	119-93-7	8270 (3520)	2.5	20.00	NA	Not Available	NA
3-Methylcholanthrene	56-49-5	8270 (3520)	1.4	10.00	NA	Not Available	NA
3-Nitroaniline	99-09-2	8270 (3520)	0.69	50.00	NA	Not Available	NA
4,6-Dinitro-2-methylphenol	534-52-1	8270 (3520)	1.0	50.00	0.01	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
4-Aminobiphenyl	92-67-1	8270 (3520)	0.68	10.00	NA	Not Available	NA
4-Bromophenyl phenyl ether	101-55-3	8270 (3520)	0.85	10.00	NA	Not Available	NA
4-Chloro-3-methyl-phenol (MS)	59-50-7	8270 (3520)	0.55	10.00	NA	Not Available	NA
4-Chloroaniline	106-47-8	8270 (3520)	0.52	20.00	28.00	IEPA TACO Class I	Yes
4-Chlorophenylphenyl ether	7005-72-3	8270 (3520)	0.56	10.00	NA	Not Available	NA
4-Nitroaniline	100-01-6	8270 (3520)	0.85	50.00	NA	Not Available	NA
4-Nitrophenol (MS)	100-02-7	8270 (3520)	3.4	50.00	290.00	Region 6 (Tap Water)	Yes
4-Nitroquinoline-1-oxide	56-57-5	8270 (3520)	5.0	20.00	NA	Not Available	NA
5-Nitro-o-toluidine	99-55-8	8270 (3520)	1.2	10.00	NA	Not Available	NA
7,12-Dimethylbenz(a)anthracene	57-97-6	8270 (3520)	1.3	10.00	NA	Not Available	NA
a,a-Dimethylphenethylamine	122-09-8	8270 (3520)	10	2000.00	NA	Not Available	NA
Acenaphthene (MS)	83-32-9	8270 (3520)	0.64	10.00	420.00	IEPA TACO Class I	Yes
Acenaphthylene	208-96-8	8270 (3520)	0.58	10.00	NA	Not Available	NA
Acetophenone	98-86-2	8270 (3520)	0.83	10.00	NA	Not Available	NA
Aniline	62-53-3	8270 (3520)	0.65	20.00	23.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Anthracene	120-12-7	8270 (3520)	0.88	10.00	2100.00	IEPA TACO Class I	Yes

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Aramite	140-57-8	8270 (3520)	1.7	10.00	NA	Not Available	NA
Benzidine	92-87-5	8270 (3520)	14	80.00	NA	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Benzo(a)anthracene	56-55-3	8270 (3520)	0.58	10.00	0.13	IEPA TACO Class I	No
Benzo(a)pyrene	50-32-8	8270 (3520)	1.0	10.00	0.20	IEPA TACO Class I	No
Benzo(b)fluoranthene	205-99-2	8270 (3520)	0.78	10.00	0.18	IEPA TACO Class I	No
Benzo(g,h,i)perylene	191-24-2	8270 (3520)	1.0	10.00	0.21	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Benzo(k)fluoranthene	207-08-9	8270 (3520)	0.73	10.00	0.17	IEPA TACO Class I	No
Benzoic acid	65-85-0	8270 (3520)	2.5	50.00	28000.00	IEPA TACO Class I	Yes
Benzyl alcohol	100-51-6	8270 (3520)	0.80	10.00	2100.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Bis(2-chloroethoxy) methane	111-91-1	8270 (3520)	0.78	10.00	NA	Not Available	NA
Bis(2-chloroethyl) ether	111-44-4	8270 (3520)	0.72	10.00	10.00	IEPA TACO Class I	Yes
Bis(2-chloroisopropyl)ether (2,2-Oxybis(1-chloropropane))	111-44-4	8270 (3520)	0.82	10.00	10.00	IEPA TACO Class I	Yes
Bis(2-ethylhexyl) phthalate	117-81-7	8270 (3520)	0.98	10.00	6.00	IEPA TACO Class I	No
Butyl benzyl phthalate	85-68-7	8270 (3520)	0.70	10.00	1400.00	IEPA TACO Class I	Yes
Carbazole	86-74-8	8270 (3520)	0.82	10.00	3.40	Region 9 (Tap Water)	No
Chrysene	218-01-9	8270 (3520)	1.0	10.00	1.50	IEPA TACO Class I	No
Diallate	2303-16-4	8270 (3520)	1.6	10.00	NA	Not Available	NA
Dibenz(a,h)anthracene	53-70-3	8270 (3520)	1.0	10.00	0.30	IEPA TACO Class I	No
Dibenzofuran	132-64-9	8270 (3520)	0.69	10.00	28.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Diethyl phthalate	84-66-2	8270 (3520)	0.59	10.00	5600.00	IEPA TACO Class I	Yes
Dimethoate	60-51-5	8270 (3520)	0.64	10.00	NA	Not Available	NA
Dimethylphthalate	131-11-3	8270 (3520)	0.59	10.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Di-n-butyl phthalate	84-74-2	8270 (3520)	0.82	10.00	4000.00	IEPA TACO Class I	Yes
Di-n-octyl phthalate	117-84-0	8270 (3520)	0.80	10.00	140.00	IEPA TACO Class I	Yes
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	88-85-7	8270 (3520)	2.5	10.00	7.00	IEPA TACO Class I	No
Diphenylamine/ Nitrosodiphenylamine	122-39-4	8270 (3520)	0.80	10.00	175.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Disulfoton	298-04-4	8270 (3520)	0.78	10.00	0.28	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Ethyl methanesulfonate	62-50-0	8270 (3520)	0.90	10.00	NA	Not Available	NA
Ethyl parathion	56-38-2	8270 (3520)	0.89	10.00	168.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Famphur	52-85-7	8270 (3520)	2.2	10.00	NA	Not Available	NA
Fluoranthene	206-44-0	8270 (3520)	1.0	10.00	280.00	IEPA TACO Class I	Yes
Fluorene	86-73-7	8270 (3520)	0.55	10.00	280.00	IEPA TACO Class I	Yes
Hexachlorobenzene	118-74-1	8270 (3520)	1.1	10.00	0.06	IEPA TACO Class I	No
Hexachlorobutadiene	87-68-3	8270 (3520)	1.0	10.00	1.40	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Hexachlorocyclopentadiene	77-47-4	8270 (3520)	5.0	10.00	50.00	IEPA TACO Class I	Yes
Hexachloroethane	67-72-1	8270 (3520)	1.0	10.00	7.00	IEPA TACO Class I	No
Hexachlorophene	70-30-4	8270 (3520)	47	5000.00	NA	Not Available	NA
Hexachloropropene	1888-71-7	8270 (3520)	1.2	10.00	NA	Not Available	NA
Indeno(1,2,3-cd)pyrene	193-39-5	8270 (3520)	0.62	10.00	0.43	IEPA TACO Class I	No
Isophorone	78-59-1	8270 (3520)	0.67	10.00	1400.00	IEPA TACO Class I	Yes
Isosafrole	120-58-1	8270 (3520)	0.96	10.00	NA	Not Available	NA
m-Dinitrobenzene	99-65-0	8270 (3520)	0.68	10.00	0.70	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Methapyrilene	91-80-5	8270 (3520)	1.9	2000.00	NA	Not Available	NA
Methyl parathion	298-00-0	8270 (3520)	0.78	10.00	NA	Not Available	NA
Methylmethanesulfonate	66-27-3	8270 (3520)	0.85	10.00	NA	Not Available	NA
Naphthalene	91-20-3	8270 (3520)	0.76	10.00	140.00	IEPA TACO Class I	Yes
Nitrobenzene	98-95-3	8270 (3520)	0.93	10.00	3.50	IEPA TACO Class I	No
Nitrosodiphenylamine/ Diphenylamine	86-30-6, 122-39-4	8270 (3520)	0.80	10.00	175.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
N-Nitrosodiethylamine	55-18-5	8270 (3520)	0.82	10.00	NA	Not Available	NA
N-Nitrosodimethylamine	62-75-9	8270 (3520)	1.1	10.00	NA	Not Available	NA
N-Nitrosodi-n-butylamine	924-16-3	8270 (3520)	0.78	10.00	NA	Not Available	NA
N-Nitrosodi-n-propylamine(MS)	621-64-7	8270 (3520)	0.62	10.00	1.80	IEPA TACO Class I	No
N-Nitrosomethylethylamine	10595-95-6	8270 (3520)	2.6	10.00	NA	Not Available	NA
N-Nitrosomorpholine	59-89-2	8270 (3520)	1.1	10.00	NA	Not Available	NA
N-Nitrosopiperidine	100-75-4	8270 (3520)	1.2	10.00	NA	Not Available	NA
N-Nitrosopyrrolidine	930-55-2	8270 (3520)	0.57	10.00	NA	Not Available	NA
o,o,o-Triethyl-phosphorothioate	126-68-1	8270 (3520)	1.3	10.00	NA	Not Available	NA
o-Toluidine	95-53-4	8270 (3520)	0.58	10.00	NA	Not Available	NA
p-(Dimethylamino)azobenzene	60-11-7	8270 (3520)	1.2	10.00	NA	Not Available	NA
Pentachlorobenzene	608-93-5	8270 (3520)	1.0	10.00	NA	Not Available	NA
Pentachloronitrobenzene	82-68-8	8270 (3520)	1.2	10.00	21.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Pentachlorophenol (MS)	87-86-5	8270 (3520)	2.5	50.00	1.00	IEPA TACO Class I	No
Phenacetin	62-44-2	8270 (3520)	0.82	10.00	NA	Not Available	NA
Phenanthrene	85-01-8	8270 (3520)	0.74	10.00	0.21	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Phenol (MS)	108-95-2	8270 (3520)	0.99	10.00	100.00	IEPA TACO Class I	Yes

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Phorate	298-02-2	8270 (3520)	0.83	10.00	14.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
p-Phenylenediamine	106-50-3	8270 (3520)	37	2000.00	NA	Not Available	NA
Pronamide	23950-58-5	8270 (3520)	0.72	10.00	NA	Not Available	NA
Pyrene(MS)	129-00-0	8270 (3520)	1.0	10.00	210.00	IEPA TACO Class I	Yes
Pyridine	110-86-1	8270 (3520)	0.81	50.00	7.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Safrole	94-59-7	8270 (3520)	0.99	10.00	NA	Not Available	NA
Sulfotepp	3689-24-5	8270 (3520)	0.70	10.00	NA	Not Available	NA
Thionazin	297-97-2	8270 (3520)	0.99	10.00	NA	Not Available	NA
Surrogates							
2-Fluorobiphenyl	321-60-8	8270 (3520)	NA	NA	NA	Not Available	NA
2-Fluorophenol	367-12-4	8270 (3520)	NA	NA	NA	Not Available	NA
Nitrobenzene-d5	98-95-3	8270 (3520)	NA	NA	3.50	IEPA TACO Class I	No
Phenol-d5	108-95-2	8270 (3520)	NA	NA	100.00	IEPA TACO Class I	No
Terphenyl-d14	26140-60-3	8270 (3520)	NA	NA	NA	Not Available	NA
2,4,6-Tribromophenol		8270 (3520)	NA	NA	NA	Not Available	NA
Non-Routine Analytes							
Atrazine	1912-24-9	8270 (3520)	0.89	10.00	3.00	IEPA TACO Class I	No
Benzaldehyde	100-52-7	8270 (3520)	1.0	10.00	700.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
1,1-Biphenyl (1,1-Diphenyl)		8270 (3520)	0.68	10.00	NA	Not Available	NA
Caprolactam	105-60-2	8270 (3520)	0.84	10.00	3500.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
2,3-Dimethylphenol	526-75-0	8270 (3520)	0.86	10.00	NA	Not Available	NA
2,5-Dimethylphenol	95-87-4	8270 (3520)	0.93	10.00	NA	Not Available	NA
2,6-Dimethylphenol	576-26-1	8270 (3520)	0.69	10.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
3,4-Dimethylphenol	95-65-8	8270 (3520)	0.79	10.00	NA	Not Available	NA
2,5-Dinitrophenol	329-71-5	8270 (3520)	0.99	50.00	NA	Not Available	NA
3-Nitrophenol	554-84-7	8270 (3520)	0.78	10.00	NA	Not Available	NA
Phenyl ether (Diphenyl oxide)	101-84-8	8270 (3520)	1.0	10.00	NA	Not Available	NA
1,2,3,5-Tetrachlorobenzene	634-90-2	8270 (3520)	0.75	10.00	NA	Not Available	NA
1,2,3-Trichlorobenzene	87-61-6	8270 (3520)	0.59	10.00	70.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
1,3,5-Trichlorobenzene	108-70-3	8270 (3520)	0.62	10.00	NA	Not Available	NA

Table 4.2
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Metals						
Aluminum	6010 (3010)	1	75-125	<=20	30	200
Antimony	6010 (3010)	1	75-125	<=20	5.1	20
Arsenic	6010 (3010)	1	75-125	<=20	5.2	10
Barium	6010 (3010)	1	75-125	<=20	1.2	10
Beryllium	6010 (3010)	1	75-125	<=20	0.14	4.0
Cadmium	6010 (3010)	1	75-125	<=20	1.0	5.0
Calcium	6010 (3010)	1	75-125	<=20	50	500
Chromium	6010 (3010)	1	75-125	<=20	0.95	10
Cobalt	6010 (3010)	1	75-125	<=20	1.3	10
Copper	6010 (3010)	1	75-125	<=20	1.7	20
Iron	6010 (3010)	1	75-125	<=20	28	50
Lead	6010 (3010)	1	75-125	<=20	3.5	5.0
Magnesium	6010 (3010)	1	75-125	<=20	9.8	500
Manganese	6010 (3010)	1	75-125	<=20	2.0	10
Nickel	6010 (3010)	1	75-125	<=20	1.5	40
Potassium	6010 (3010)	1	75-125	<=20	15	1000
Selenium	6010 (3010)	1	75-125	<=20	5.4	10
Silver	6010 (3010)	1	75-125	<=20	1.0	10
Sodium	6010 (3010)	1	75-125	<=20	300	1000
Thallium	6010 (3010)	1	75-125	<=20	11	25
Tin	6010 (3010)	1	75-125	<=20	6.3	50
Vanadium	6010 (3010)	1	75-125	<=20	1.4	10
Zinc	6010 (3010)	1	75-125	<=20	3.0	20
Mercury (CVAA)	7470	1	80-120	<=20	0.080	0.20
Polychlorinated Biphenyls as Aroclors by GC/EC						
Monochlorobiphenyls	680	24	18-97	<=40	0.010	0.10
Dichlorobiphenyls	680	24	18-97	<=40	0.012	0.10
Trichlorobiphenyls	680	24	23-98	<=40	0.012	0.10
Tetrachlorobiphenyls	680	24	25-100	<=40	0.023	0.20
Pentachlorobiphenyls	680	24	39-100	<=40	0.026	0.20
Hexachlorobiphenyls	680	24	33-110	<=40	0.024	0.20
Heptachlorobiphenyls	680	24	43-102	<=40	0.037	0.30
Octachlorobiphenyls	680	24	44-104	<=40	0.036	0.30
Nonachlorobiphenyls	680	24	44-104	<=40	0.062	0.50
Decachlorobiphenyl	680	24	44-104	<=40	0.084	0.50
Surrogate						
Decachlorobiphenyl-13C12	680	24	44-104	NA	NA	NA
Chlorinated Pesticides in Groundwater by GC/EC						
4,4'-DDD	8081 (3520)	1	59-141	<=40	0.011	0.10
4,4'-DDE	8081 (3520)	1	44-117	<=40	0.012	0.10
4,4'-DDT (MS)	8081 (3520)	1	39-140	<=40	0.014	0.10
Aldrin (MS)	8081 (3520)	1	37-120	<=40	0.0053	0.050
alpha BHC	8081 (3520)	1	31-112	<=40	0.0086	0.050
alpha Chlordane	8081 (3520)	1	45-120	<=40	0.0068	0.050
beta BHC	8081 (3520)	1	19-131	<=40	0.0057	0.050
Chlorobenzilate	8081 (3520)	1	35-155	<=40	0.19	0.50

Table 4.2
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Chlorinated Pesticides in Groundwater by GC/EC						
delta BHC	8081 (3520)	1	39-123	<=40	0.0098	0.050
Dieldrin (MS)	8081 (3520)	1	38-136	<=40	0.010	0.10
Endosulfan I	8081 (3520)	1	40-119	<=40	0.0064	0.050
Endosulfan II	8081 (3520)	1	40-123	<=40	0.011	0.10
Endosulfan sulfate	8081 (3520)	1	54-132	<=40	0.010	0.10
Endrin (MS)	8081 (3520)	1	34-146	<=40	0.010	0.10
Endrin aldehyde	8081 (3520)	1	33-142	<=40	0.017	0.10
Endrin ketone	8081 (3520)	1	37-165	<=40	0.016	0.10
gamma BHC (Lindane) (MS)	8081 (3520)	1	27-115	<=40	0.0055	0.050
gamma Chlordane	8081 (3520)	1	55-123	<=40	0.013	0.050
Heptachlor (MS)	8081 (3520)	1	35-121	<=40	0.0076	0.050
Heptachlor epoxide	8081 (3520)	1	49-120	<=40	0.015	0.050
Isodrin	8081 (3520)	1	24-214	<=40	0.0082	0.050
Kepone	8081 (3520)	1	D-105	<=100	0.12	1.0
Methoxychlor	8081 (3520)	1	53-167	<=40	0.024	0.50
Toxaphene	8081 (3520)	1	39-137	<=40	0.88	5.0
Surrogates						
Non-routine Compounds						
2,4'-DDD	8081 (3520)	1	59-141	<=40	0.073	0.10
2,4'-DDE	8081 (3520)	1	44-117	<=40	0.068	0.10
2,4'-DDT	8081 (3520)	1	39-140	<=40	0.071	0.10
Chlorinated Herbicides by GC/EC						
2,4,5-T (MS)	8151	1	38-138	<=40	0.080	0.50
2,4,5-TP (Silvex) (MS)	8151	1	44-118	<=40	0.033	0.50
2,4-D (MS)	8151	1	45-146	<=40	0.11	0.50
2,4-DB	8151	1	35-140	<=40	0.057	0.50
Dalapon	8151	1	48-187	<=40	0.24	10
Dicamba	8151	1	60-113	<=40	0.048	0.50
Dichlorprop	8151	1	43-106	<=40	0.030	0.50
Dinoseb	8151	1	23-117	<=40	0.40	6.0
MCPA	8151	1	47-131	<=40	12	120
MCPP	8151	1	27-150	<=40	10	120
Pentachlorophenol	8151	1	46-144	<=40	0.18	0.25
Volatiles in Groundwater by GC/MS						
1,1,1,2-Tetrachloroethane	8260 (5030)	1	62-107	<=30	0.53	1.0
1,1,1-Trichloroethane	8260 (5030)	1	70-132	<=30	0.79	1.0
1,1,2,2-Tetrachloroethane	8260 (5030)	1	71-127	<=30	0.21	1.0
1,1,2-Trichloroethane	8260 (5030)	1	75-122	<=30	0.37	1.0
1,1-Dichloroethane	8260 (5030)	1	70-127	<=30	0.56	1.0
1,1-Dichloroethene (MS)	8260 (5030)	1	64-132	<=30	0.93	1.0
1,2,3-Trichloropropane	8260 (5030)	1	60-147	<=30	0.44	1.0
1,2,4-Trichlorobenzene	8260 (5030)	1	48-131	<=30	0.28	1.0
1,2-Dibromo-3-chloropropane	8260 (5030)	1	14-147	<=50	0.65	1.0
1,2-Dibromoethane	8260 (5030)	1	60-118	<=30	0.34	1.0
1,2-Dichlorobenzene	8260 (5030)	1	71-125	<=30	0.21	1.0
1,2-Dichloroethane	8260 (5030)	1	68-130	<=30	0.28	1.0

Table 4.2
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Volatiles in Groundwater by GC/MS						
1,2-Dichloroethenes, Total (sum of cis- and trans- isomers)	8260 (5030)	1	70-130	<=30	1.2	2.0
1,2-Dichloropropane	8260 (5030)	1	74-123	<=30	0.26	1.0
1,3-Dichlorobenzene	8260 (5030)	1	70-125	<=30	0.28	1.0
1,4-Dichlorobenzene	8260 (5030)	1	65-127	<=30	0.44	1.0
2-Butanone (methyl ethyl ketone-MEK)	8260 (5030)	1	51-142	<=30	0.72	10
2-Chloro-1,3-butadiene (Chloroprene)	8260 (5030)	1	70-130	<=30	0.40	1.0
2-Hexanone	8260 (5030)	1	58-139	<=30	0.39	10
3-Chloropropene (Allyl chloride)	8260 (5030)	1	D-200	<=100	0.35	1.0
4-Methyl-2-pentanone (MIBK)	8260 (5030)	1	62-130	<=30	0.45	10
Acetone	8260 (5030)	1	20-183	<=50	7.3	25
Acetonitrile	8260 (5030)	1	71-158	<=30	0.23	40
Acrolein	8260 (5030)	1	40-91	<=30	12	20
Acrylonitrile	8260 (5030)	1	46-144	<=30	3.2	20
Benzene (MS)	8260 (5030)	1	74-122	<=30	0.54	1.0
Bromodichloromethane	8260 (5030)	1	74-128	<=30	0.42	1.0
Bromoform	8260 (5030)	1	64-132	<=30	0.36	1.0
Bromomethane	8260 (5030)	1	21-176	<=50	0.93	1.0
Carbon disulfide	8260 (5030)	1	60-130	<=30	0.75	1.0
Carbon tetrachloride	8260 (5030)	1	64-137	<=30	0.91	1.0
Chlorobenzene (MS)	8260 (5030)	1	75-123	<=30	0.41	1.0
Chloroethane	8260 (5030)	1	40-171	<=50	0.89	1.0
Chloroform	8260 (5030)	1	74-124	<=30	0.52	1.0
Chloromethane	8260 (5030)	1	51-133	<=50	0.53	1.0
cis-1,2-Dichloroethene	8260 (5030)	1	69-126	<=30	0.55	1.0
cis-1,3-Dichloropropene	8260 (5030)	1	76-126	<=30	0.25	1.0
Dibromochloromethane	8260 (5030)	1	75-126	<=30	0.40	1.0
Dibromomethane	8260 (5030)	1	70-130	<=30	0.33	1.0
Dichlorodifluoromethane	8260 (5030)	1	70-130	<=50	0.73	1.0
Ethyl methacrylate	8260 (5030)	1	58-101	<=30	0.33	1.0
Ethylbenzene	8260 (5030)	1	77-123	<=30	0.62	1.0
Hexachlorobutadiene	8260 (5030)	1	58-133	<=30	0.50	1.0
Iodomethane	8260 (5030)	1	34-116	<=30	0.96	5.0
Isobutyl alcohol	8260 (5030)	1	39-132	<=30	13	40
m&p-Xylene	8260 (5030)	1	74-123	<=30	1.3	2.0
Methacrylonitrile	8260 (5030)	1	65-110	<=30	9.1	20
Methylene chloride	8260 (5030)	1	67-128	<=30	0.44	5.0
Methylmethacrylate	8260 (5030)	1	57-120	<=30	0.36	1.0
Naphthalene	8260 (5030)	1	58-143	<=30	0.12	5.0
o-Xylene	8260 (5030)	1	76-122	<=30	0.49	1.0
Pentachloroethane	8260 (5030)	1	1-200	<=100	1.0	5.0
Propionitrile (ethylcyanide)	8260 (5030)	1	72-121	<=30	10	20
Styrene	8260 (5030)	1	75-125	<=30	0.42	1.0
Tetrachloroethene	8260 (5030)	1	70-133	<=30	0.75	1.0
Toluene (MS)	8260 (5030)	1	75-122	<=30	0.62	1.0
trans-1,2-Dichloroethene	8260 (5030)	1	67-130	<=30	0.80	1.0
trans-1,3-Dichloropropene	8260 (5030)	1	75-126	<=30	0.36	1.0

Table 4.2
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Volatiles in Groundwater by GC/MS						
trans-1,4-Dichloro-2-butene	8260 (5030)	1	26-131	<=50	0.80	2.0
Trichloroethene(MS)	8260 (5030)	1	75-122	<=30	0.71	1.0
Trichlorofluoromethane	8260 (5030)	1	74-165	<=50	0.96	1.0
Vinyl acetate	8260 (5030)	1	47-150	<=30	0.70	2.0
Vinyl chloride	8260 (5030)	1	59-136	<=50	0.92	1.0
Xylenes (total)	8260 (5030)	1	77-121	<=30	1.6	2.0
Semivolatiles in Groundwater by GC/MS						
1,2,4,5-Tetrachlorobenzene	8270 (3520)	1	17-127	<=40	1.2	10
1,2,4-Trichlorobenzene (MS)	8270 (3520)	1	46-99	<=40	0.54	10
1,2-Dichlorobenzene	8270 (3520)	1	42-98	<=40	1.0	10
1,3,5-Trinitrobenzene	8270 (3520)	1	10-123	<=40	0.62	10
1,3-Dichlorobenzene	8270 (3520)	1	38-97	<=40	0.55	10
1,4-Dichlorobenzene(MS)	8270 (3520)	1	40-92	<=40	0.52	10
1,4-Dioxane	8270 (3520)	1	22-134	<=40	2.3	10
1,4-Naphthoquinone	8270 (3520)	1	23-49	<=40	0.67	10
1-Naphthylamine	8270 (3520)	1	10-127	<=40	1.2	10
2,3,4,6-Tetrachlorophenol	8270 (3520)	1	10-151	<=40	0.47	10
2,4,5-Trichlorophenol	8270 (3520)	1	62-119	<=40	0.63	10
2,4,6-Trichlorophenol	8270 (3520)	1	61-118	<=40	0.70	10
2,4-Dichlorophenol	8270 (3520)	1	62-112	<=40	0.66	10
2,4-Dimethylphenol	8270 (3520)	1	51-111	<=40	1.0	10
2,4-Dinitrophenol	8270 (3520)	1	13-176	<=40	5.0	50
2,4-Dinitrotoluene (MS)	8270 (3520)	1	45-140	<=40	0.56	10
2,6-Dichlorophenol	8270 (3520)	1	17-125	<=40	0.69	10
2,6-Dinitrotoluene	8270 (3520)	1	65-124	<=40	0.57	10
2-Chloronaphthalene	8270 (3520)	1	58-111	<=40	0.62	10
2-Chlorophenol (MS)	8270 (3520)	1	54-106	<=40	0.72	10
2-Methyl phenol	8270 (3520)	1	57-110	<=40	0.70	10
2-Methylnaphthalene	8270 (3520)	1	51-110	<=40	0.53	10
2-Naphthylamine	8270 (3520)	1	10-119	<=40	0.64	10
2-Nitroaniline	8270 (3520)	1	60-122	<=40	0.69	50
2-Nitrophenol	8270 (3520)	1	59-114	<=40	0.73	10
2-Picoline	8270 (3520)	1	35-102	<=40	0.87	10
3- and 4-Methyl phenol	8270 (3520)	1	49-114	<=40	0.66	10
3,3'-Dichlorobenzidine	8270 (3520)	1	29-101	<=40	1.0	20
3,3'-Dimethylbenzidine	8270 (3520)	1	10-200	<=40	2.5	20
3-Methylcholanthrene	8270 (3520)	1	10-129	<=40	1.4	10
3-Nitroaniline	8270 (3520)	1	46-144	<=40	0.69	50
4,6-Dinitro-2-methylphenol	8270 (3520)	1	42-155	<=40	1.0	50
4-Aminobiphenyl	8270 (3520)	1	20-105	<=40	0.68	10
4-Bromophenyl phenyl ether	8270 (3520)	1	50-112	<=40	0.85	10
4-Chloro-3-methyl-phenol (MS)	8270 (3520)	1	58-118	<=40	0.55	10
4-Chloroaniline	8270 (3520)	1	22-107	<=100	0.52	20
4-Chlorophenylphenyl ether	8270 (3520)	1	59-112	<=40	0.56	10
4-Nitroaniline	8270 (3520)	1	47-127	<=40	0.85	50
4-Nitrophenol (MS)	8270 (3520)	1	30-139	<=40	3.4	50
4-Nitroquinoline-1-oxide	8270 (3520)	1	30-130	<=40	5.0	20

Table 4.2
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (ug/L)	RL (ug/L)
Semivolatiles in Groundwater by GC/MS						
5-Nitro-o-toluidine	8270 (3520)	1	10-112	<=40	1.2	10
7,12-Dimethylbenz(a)anthracene	8270 (3520)	1	19-112	<=40	1.3	10
a,a-Dimethylphenethylamine	8270 (3520)	1	10-200	<=40	10	2000
Acenaphthene (MS)	8270 (3520)	1	49-117	<=40	0.64	10
Acenaphthylene	8270 (3520)	1	53-117	<=40	0.58	10
Acetophenone	8270 (3520)	1	54-130	<=40	0.83	10
Aniline	8270 (3520)	1	10-92	<=40	0.65	20
Anthracene	8270 (3520)	1	57-118	<=40	0.88	10
Aramite	8270 (3520)	1	10-168	<=40	1.7	10
Benzo(a)anthracene	8270 (3520)	1	55-119	<=40	0.58	10
Benzo(a)pyrene	8270 (3520)	1	36-128	<=40	1.0	10
Benzo(b)fluoranthene	8270 (3520)	1	44-130	<=40	0.78	10
Benzo(g,h,i)perylene	8270 (3520)	1	45-128	<=40	1.0	10
Benzo(k)fluoranthene	8270 (3520)	1	47-129	<=40	0.73	10
Benzyl alcohol	8270 (3520)	1	54-116	<=40	0.80	10
Bis(2-chloroethoxy) methane	8270 (3520)	1	55-115	<=40	0.78	10
Bis(2-chloroethyl) ether	8270 (3520)	1	48-108	<=40	0.72	10
Bis(2-chloroisopropyl)ether (2,2-Oxybis(1-chloropropane))	8270 (3520)	1	45-114	<=40	0.82	10
Bis(2-ethylhexyl) phthalate	8270 (3520)	1	57-125	<=40	0.98	10
Butyl benzyl phthalate	8270 (3520)	1	62-124	<=40	0.70	10
Carbazole	8270 (3520)	1	60-120	<=40	0.82	10
Chrysene	8270 (3520)	1	56-122	<=40	1.0	10
Diallate	8270 (3520)	1	24-148	<=40	1.6	10
Dibenz(a,h)anthracene	8270 (3520)	1	47-126	<=40	1.0	10
Dibenzofuran	8270 (3520)	1	65-109	<=40	0.69	10
Diethyl phthalate	8270 (3520)	1	61-115	<=40	0.59	10
Dimethoate	8270 (3520)	1	48-77	<=40	0.64	10
Dimethylphthalate	8270 (3520)	1	64-112	<=40	0.59	10
Di-n-butyl phthalate	8270 (3520)	1	58-122	<=40	0.82	10
Di-n-octyl phthalate	8270 (3520)	1	55-128	<=40	0.80	10
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	8270 (3520)	1	10-127	<=40	2.5	10
Diphenylamine/ Nitrosodiphenylamine	8270 (3520)	1	30-130	<=40	0.80	10
Disulfoton	8270 (3520)	1	36-82	<=40	0.78	10
Ethyl methanesulfonate	8270 (3520)	1	45-127	<=40	0.90	10
Ethyl parathion	8270 (3520)	1	10-75	<=40	0.89	10
Famphur	8270 (3520)	1	10-248	<=40	2.2	10
Fluoranthene	8270 (3520)	1	51-125	<=40	1.0	10
Fluorene	8270 (3520)	1	43-124	<=40	0.55	10
Hexachlorobenzene	8270 (3520)	1	60-122	<=40	1.1	10
Hexachlorobutadiene	8270 (3520)	1	43-109	<=40	1.0	10
Hexachlorocyclopentadiene	8270 (3520)	1	1-84	<=100	5.0	10
Hexachloroethane	8270 (3520)	1	35-89	<=40	1.0	10
Hexachlorophene	8270 (3520)	1	1-253	<=100	47	5000
Hexachloropropene	8270 (3520)	1	10-117	<=40	1.2	10
Indeno(1,2,3-cd)pyrene	8270 (3520)	1	40-132	<=40	0.62	10
Isophorone	8270 (3520)	1	60-113	<=40	0.67	10

Table 4.2
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (ug/L)	RL (ug/L)
Semivolatiles in Groundwater by GC/MS						
Isosafrole	8270 (3520)	1	26-127	<=40	0.96	10
m-Dinitrobenzene	8270 (3520)	1	10-118	<=40	0.68	10
Methapyrilene	8270 (3520)	1	10-86	<=40	1.9	2000
Methyl parathion	8270 (3520)	1	10-82	<=40	0.78	10
Methylmethanesulfonate	8270 (3520)	1	34-136	<=40	0.85	10
Naphthalene	8270 (3520)	1	42-108	<=40	0.76	10
Nitrobenzene	8270 (3520)	1	57-110	<=40	0.93	10
Nitrosodiphenylamine/ Diphenylamine	8270 (3520)	1	30-130	<=40	0.80	10
N-Nitrosodiethylamine	8270 (3520)	1	50-99	<=40	0.82	10
N-Nitrosodimethylamine	8270 (3520)	1	50-137	<=40	1.1	10
N-Nitrosodi-n-butylamine	8270 (3520)	1	30-123	<=40	0.78	10
N-Nitrosodi-n-propylamine(MS)	8270 (3520)	1	49-135	<=40	0.62	10
N-Nitrosomethylethylamine	8270 (3520)	1	10-279	<=40	2.6	10
N-Nitrosomorpholine	8270 (3520)	1	17-151	<=40	1.1	10
N-Nitrosopiperidine	8270 (3520)	1	21-156	<=40	1.2	10
N-Nitrosopyrrolidine	8270 (3520)	1	52-91	<=40	0.57	10
o,o,o-Triethyl-phosphorothioate	8270 (3520)	1	46-138	<=40	1.3	10
o-Toluidine	8270 (3520)	1	10-129	<=40	0.58	10
p-(Dimethylamino)azobenzene	8270 (3520)	1	10-113	<=40	1.2	10
Pentachlorobenzene	8270 (3520)	1	26-127	<=40	1.0	10
Pentachloronitrobenzene	8270 (3520)	1	10-157	<=40	1.2	10
Pentachlorophenol (MS)	8270 (3520)	1	44-132	<=40	2.5	50
Phenacetin	8270 (3520)	1	18-104	<=40	0.82	10
Phenanthrene	8270 (3520)	1	58-120	<=40	0.74	10
Phenol (MS)	8270 (3520)	1	46-106	<=40	0.99	10
Phorate	8270 (3520)	1	36-113	<=40	0.83	10
p-Phenylenediamine	8270 (3520)	1	10-177	<=40	37	2000
Pronamide	8270 (3520)	1	18-122	<=40	0.72	10
Pyrene(MS)	8270 (3520)	1	49-135	<=40	1.0	10
Pyridine	8270 (3520)	1	10-178	<=40	0.81	50
Safrole	8270 (3520)	1	36-114	<=40	0.99	10
Sulfotepp	8270 (3520)	1	30-117	<=40	0.70	10
Thionazin	8270 (3520)	1	42-93	<=40	0.99	10
Non-Routine Analytes						
Atrazine	8270 (3520)	1	40-132	<=50	0.89	10
Benzaldehyde	8270 (3520)	1	30-150	<=50	1.0	10
1,1-Biphenyl (1,1-Diphenyl)	8270 (3520)	1	53-118	<=50	0.68	10
Caprolactam	8270 (3520)	1	30-150	<=50	0.84	10
3-Nitrophenol	8270 (3520)	1	30-130	<=50	0.78	10

STL	REFERENCES AND NOTES FOR WATER PARAMETERS

A "non-routine analyte" is an analyte that requires advance notice prior to arrival in the laboratory. In general, at least one week notice is required to ensure that the laboratory has sufficient instrument capacity, standards, and reagents to adequately process the samples.

Accuracy data are presented as recoveries for spikes or surrogates. For routine analysis of organics using SW-846 methods, percent recoveries are evaluated only on the subset spike compound lists specified by the methods unless noted in a pre-project plan or QAPP. An (MS) following the parameter name designates the routine matrix and laboratory control spike compounds. Precision data are presented as relative percent difference (% RPD) and are advisory; i.e., not used for laboratory control. Since reportable levels (above detection limit) for most of the organic parameters may not be detected in all environmental samples, precision is usually based on duplicate spike data and evaluated according to method requirements.

Accuracy and precision control limits are primarily derived from in-house laboratory data. Some accuracy and precision limits have been rounded to the nearest "5". In some cases, method limits may be used in lieu of in-house limits because in-house limits are broader than the method limits or are too broad to be usable.

ABBREVIATIONS USED IN THE APPENDICES

PARAMETER-refers to the compound, analyte or measurement being tested; the field of testing

METHOD-refers to the reference method used to measure the parameter

REF-a number designation that corresponds to the method references and citations

ACC-accuracy measured as percent recovery

PREC-precision measured as relative percent difference

RL-reporting limit

MDL-method detection limit

D-analyte detected (meets qualitative identification criteria)

ICP-inductively coupled (argon) plasma

ICP-MS-inductively coupled (argon) plasma coupled to a mass spectrometer

GFAA -graphite furnace atomic absorption

CVAA - cold vapor atomic absorption

IC - ion chromatography

GC/HECD - gas chromatograph equipped with a Hall electrolytic conductivity detector

GC/PID - gas chromatograph equipped with a photoionization detector

GC/FID - gas chromatograph equipped with a flame ionization detector

GC/EC - gas chromatograph equipped with an electron capture detector

GC/NPD - gas chromatograph equipped with a nitrogen-phosphorus detector

GC/MS - gas chromatrograph equipped with a mass spectrometer

HPLC - high performance liquid chromatography

STL

REFERENCES AND NOTES FOR WATER PARAMETERS

REF #	REFERENCE
1	Test Methods for Evaluating Solid Waste, Third Edition, SW-846 (including Update III) USEPA Office of Solid Waste and Emergency Response, Washington, DC.
2	EPA 600/4-79-020: Methods for Chemical Analysis of Water and Wastes; U.S. EPA Office of Research and Development, Cincinatti, OH, March 1983.
3	EPA 600/R-94/111: "Methods for the Determination of Metals in Environmental Samples" May 1994, Supplement 1.
4	Standard Methods for the Examination of Water and Wastewater, 18th Edition, American Public Health Association Washington, DC.
5	EPA 200.9: Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Adsorption Spectrometry; Revision 1.2, April 1991; EPA-EMSL
6	Laboratory SOP ME72: *Silica: Preparation and Analysis*
7	Laboratory SOP GE115: *Ion Chromatography*
8	Florida Department of Environmental Regulation; Chemistry Laboratory Methods Manual, Tallahassee. "Calculation of Un-ionized Ammonia in Fresh Water" Revision 1, 10/03/83.
9	ASTM Method D 4282-89 "Standard Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion." (reapproved 1994)
10	Method 1650: Absorbable Organic Halides by Adsorption in Microcoulometric Titrations; Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Water and Wastewater: U.S. EPA Office of Water, Engineering; and Analysis Division, Washington, D.C.
11	HACH Method 8141, Hydrazine by p-Dimethylaminobenzaldehyde Method. Adapted from ASTM Manual of Industrial Wastes, D1385-78, 376 (1979)
12	EPA/CE-81-1 Technical Report, May 1981: Environmental Protection Agency/Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material; Procedures for Handling and Chemical Analysis of Sediment and Water Samples.
13	Laboratory SOP GE27: Calculation of Total and Organic Nitrogen
14	Method 1664: N-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons), EPA-821-B-94-004b, April 1995.
15	Method 314.0: Determination of Perchlorate in Drinking Water Using Ion Chromatography, Revision 1.0, November 1999. USEPA Office of Water.
16	American Society for Testing and Materials (ASTM) Method D1498-76 (re-approved in 1981); Laboratory SOP BA72: Oxidation-Reduction Potential (ORP)
17	EPA 600/4-88-039: Methods for the Determination of Organic Compounds in Drinking Water, December, 1988, Revised July, 1991. Methods for the Determination of Organic Compounds in Drinking Water, Supplement I, July 1990. Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, August 1992
18	Code of Federal Regulations, Title 40, Part 136 ; U.S. Government Printing Office: Washington, DC, July 1, 1997.
19	Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater: EPA Method 614, February 1982.
20	Determination of Organophosphorus Pesticides in Wastewater: EPA Method 614.1

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REFERENCES AND NOTES FOR WATER PARAMETERS

REF #	REFERENCE
21	Determination of Organophosphorus Pesticides in Industrial and Municipal Wastewater: EPA Method 622 ; January 1982.
22	Determination of Thiophosphate Pesticides in Industrial and Municipal Wastewater: EPA Method 622.1; January 1982.
23	Determination of Chlorinated Herbicides in Industrial and Municipal Wastewater. EPA Method 615, January 1982
24	Method 680: Determination of Pesticides and PCBs in Water and Soil/Sediment by GC/MS U.S. EPA, Washington, D.C..
25	Method 1653: Chlorinated Phenolics in Water by In-situ Acetylation/GC/MS Determination, Method cp. 86.01, National Council of the Paper Industry for Air and Stream Improvement, Inc., 260 Madison Avenue, NY 10016 (July 1986)
26	Method for the Determination of Gasoline Range Organics , State of Tennessee Department of Environment and Conservation, Division of Underground Storage Tanks
27	FL-PRO Method, "Method for Determination of Petroleum Range Organics," FL DEP, Revision 1, November 1, 1995.
28	Method for the Determination of Extractable Petroleum Hydrocarbons by GC/FID ; State of Tennessee Department of Environment and Conservation. Effective May 1, 1999.
29	TNRCC Method for Total Petroleum Hydrocarbons, TNRCC Method 1005 (Revision 03, 6/1/01), State of Texas Method.
30	"NCASI Method DI/HAPS-99.01 Selected HAPS in Condensates by GC/FID. NCASI Southern Regional Center May 2000. National Council for Air and Stream Improvement, Inc., 2000. Methods Manual.
31	"NCASI Method DI/MEOH-94.03 Methanol in Process Liquids by GC/FID" NCASI Southern Regional Center May 2000. National Council for Air and Stream Improvement, Inc., 2000. Methods Manual.
32	RSKSOP 175: Dissolved Oxygen and Methane in Water by GC Headspace Equilibration Technique by Kampbell and Wilson, USEPS, March 1989. Laboratory SOP AR30: *Dissolved Gases in Water*
33	Method 200.8: Determination of Trace Elements in Water and Wastes by ICP/MS. Revision 5.4 (EMMC Version), USEPA Office of Research and Development (1994)
34	California LUFT (Leaking Underground Fuel Tanks) Method; LUFT Manual: Guidelines for Assessment, Cleanup, and Underground Storage Tank Closure. State of California LUFT Task Force, May 1988.

Table 4.3 Recommended Sample Containers, Preservation, and Hold Times for Parameters Measured in Wastewater and Groundwater Matrices

Volatiles by GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Volatiles	8260	3 x 40-ml G	0.3ml 1:1 hydrochloric acid	14 days

Semivolatiles by GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Base/Neutrals/Acids	8270	2 x 1-L Amb G	None (6)	7 days/40 days

PCBs by GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Polychlorinated Byphenols	680	1-L Amb G	None (6)	7 days/40 days

Organochloride Pesticides and PCBs by GC

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Pesticides	8081A	1-L Amb G	None (6)	7 days/40 days

Chlorinated Herbicides

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Herbicides	8151A	2 x 1-L Amb G	None (6)	7 days/40 days

Metals

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Metals (except Mercury)	ICP: 6010 GFAA:7000-series	250 ml P	1ml 1:1 nitric acid	6 months

EVS Groundwater Data Set

APPENDIX A
TABLE A-1 EVS GROUNDWATER DATA SET
FOR FIGURES 2-1 THROUGH 2-7, 2-8, AND B-1 THROUGH B-33

Point ID	Easting	Northing	Surface Elevation (ft MSL)	Depth (ft bgs)	Depth Elevation (ft MSL)	MCB Concentration (ppm)	DCB Concentration (ppm)	Benzene Concentration (ppm)	Chlorobenzene Concentration (ppm)	Chloroaniline Concentration (ppm)	Total Chlorophenols Concentration (ppm)	Total Dichlorobenzene Concentration (ppm)	2,4-Dichlorophenol Concentration (ppm)	Phenol Concentration (ppm)	2,4,6-Trichlorophenol Concentration (ppm)	Total Methylphenols Concentration (ppm)	Acetone Concentration (ppm)	BTEX Concentration (ppm)
AA-GWM-S1	2292140.98	704253.78	422	139.5	282.5	3000	103	56	3000	5	38.3	103	2.3	N.D.	N.D.	N.D.	N.D.	56
AA-GWM-S1	2292140.98	704253.78	422	119.5	302.5	3700	198.5	68	3700	6.3	51.3	198.5	5.3	N.D.	N.D.	N.D.	N.D.	68
AA-GWM-S1	2292140.98	704253.78	422	99.5	322.5	2800	20.3	76	2800	12	40	20.3	N.D.	N.D.	N.D.	N.D.	N.D.	76
AA-GWM-S1	2292140.98	704253.78	422	79.5	342.5	620	N.D.	79	620	2.2	8.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	82.6
AA-GWM-S1	2292140.98	704253.78	422	59.5	362.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-GWM-S1	2292140.98	704253.78	422	39.5	382.5	2.7	N.D.	0.59	2.7	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.59
AA-GWM-S2	2292911.75	703864.9	410.5	119.5	291	6700	387	23	6700	N.D.	46	387	4	1.5	N.D.	N.D.	N.D.	23
AA-GWM-S2	2292911.75	703864.9	410.5	99.5	311	8200	195.4	18	8200	N.D.	58.3	195.4	2.3	N.D.	N.D.	N.D.	N.D.	18
AA-GWM-S2	2292911.75	703864.9	410.5	79.5	331	5100	127.9	N.D.	5100	N.D.	50.8	127.9	1.8	N.D.	N.D.	N.D.	N.D.	N.D.
AA-GWM-S2	2292911.75	703864.9	410.5	59.5	351	78	N.D.	0.62	78	N.D.	2.3	N.D.	N.D.	N.D.	N.D.	N.D.	11	0.62
AA-GWM-S2	2292911.75	703864.9	410.5	39.5	371	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-GWM-S3	2293802.69	703427.62	410.5	99.5	311	5500	203.7	1900	5500	6.2	28	203.7	1.7	40	N.D.	N.D.	N.D.	1900
AA-GWM-S3	2293802.69	703427.62	410.5	79.5	331	40000	4	24000	40000	10	38	4	N.D.	95	N.D.	N.D.	N.D.	24088
AA-GWM-S3	2293802.69	703427.62	410.5	59.5	351	100000	6.8	74000	100000	14	60	6.8	N.D.	170	N.D.	N.D.	N.D.	74000
AA-GWM-S3	2293802.69	703427.62	410.5	39.5	371	180000	14.1	81000	180000	N.D.	70	14.1	N.D.	120	N.D.	N.D.	N.D.	81000
AA-GWM-S3	2293802.69	703427.62	410.5	19.5	391	89	N.D.	N.D.	89	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-1	2291916.422	702085.598	408	120	288	400	22.2	N.D.	400	N.D.	7.7	22.2	1.2	N.D.	N.D.	N.D.	N.D.	1
AA-O-1	2291916.422	702085.598	408	116	292	900	68.3	N.D.	900	2.8	20.5	68.3	3.5	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-1	2291916.422	702085.598	408	106	302	1300	84.2	9.6	1300	4.1	22.4	84.2	3.4	N.D.	N.D.	N.D.	N.D.	9.6
AA-O-1	2291916.422	702085.598	408	96	312	1500	54	N.D.	1500	3.4	18	54	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-1	2291916.422	702085.598	408	86	322	620	4.8	N.D.	620	1.1	11	4.8	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-1	2291916.422	702085.598	408	76	332	180	3	N.D.	180	N.D.	4.8	3	N.D.	N.D.	N.D.	N.D.	N.D.	0.52
AA-O-1	2291916.422	702085.598	408	66	342	7.8	1.2	N.D.	7.8	N.D.	N.D.	1.2	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-1	2291916.422	702085.598	408	56	352	5.6	N.D.	N.D.	5.6	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
AA-O-1	2291916.422	702085.598	408	46	362	7.1	3.6	N.D.	7.1	N.D.	N.D.	3.6	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
AA-O-1	2291916.422	702085.598	408	36	372	32	10.9	N.D.	32	N.D.	N.D.	10.9	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
AA-O-1	2291916.422	702085.598	408	26	382	5.4	N.D.	0.13	5.4	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	0.13
AA-O-1	2291916.422	702085.598	408	16	392	7.8	N.D.	0.1	7.8	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	0.1
AA-O-2	2291757.411	702108.597	408	124	284	70	N.D.	0.33	70	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.91
AA-O-2	2291757.411	702108.597	408	121	287	560	9.7	N.D.	560	N.D.	8.1	9.7	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-2	2291757.411	702108.597	408	113	295	1000	53.9	4.5	1000	1.4	15.7	53.9	1.7	N.D.	N.D.	N.D.	N.D.	4.5
AA-O-2	2291757.411	702108.597	408	103	305	460	9.3	N.D.	460	N.D.	7.5	9.3	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-2	2291757.411	702108.597	408	93	315	850	11.1	2.4	850	1.4	12	11.1	N.D.	N.D.	N.D.	N.D.	N.D.	2.4
AA-O-2	2291757.411	702108.597	408	83	325	380	4.1	N.D.	380	1.3	7.6	4.1	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-2	2291757.411	702108.597	408	73	335	32	N.D.	N.D.	32	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-2	2291757.411	702108.597	408	63	345	14	N.D.	N.D.	14	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-2	2291757.411	702108.597	408	53	355	16	2.6	N.D.	16	N.D.	N.D.	2.6	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-2	2291757.411	702108.597	408	43	365	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-2	2291757.411	702108.597	408	33	375	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.77
AA-O-2	2291757.411	702108.597	408	23	385	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-2	2291757.411	702108.597	408	13	395	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	1.04
AA-O-3	2291326.5	702360.05	414	128	286	520	5.1	N.D.	520	N.D.	13	5.1	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-3	2291326.5	702360.05	414	118	296	690	9.1	N.D.	690	N.D.	15	9.1	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-3	2291326.5	702360.05	414	108	306	490	6	N.D.	490	1.2	6.8	6	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-3	2291326.5	702360.05	414	98	316	340	6.9	N.D.	340	1.3	6.8	6.9	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-3	2291326.5	702360.05	414	88	326	100	1.3	N.D.	100	N.D.	2	1.3	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-3	2291326.5	702360.05	414	78	336	12	N.D.	N.D.	12	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-3	2291326.5	702360.05	414	68	346	8.8	N.D.	N.D.	8.8	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-3	2291326.5	702360.05	414	58	356	4.8	N.D.	N.D.	4.8	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-3	2291326.5	702360.05	414	48	366	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-3	2291326.5	702360.05	414	38	376	0.81	N.D.	N.D.	0.81	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-O-3	2291326.5	702360.05	414	28	386	0.48	N.D.	N.D.	0.48	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-1	2293284.955	705504.714	408	120	288	2300	3.1	77	2300	2.8	25.9	3.1	N.D.	2.8	N.D.	N.D.	N.D.	77
AA-P-1	2293284.955	705504.714	408	114	294	4600	9.8	60	4600	7.3	46	9.8	N.D.	3.4	N.D.	N.D.	N.D.	60
AA-P-1	2293284.955	705504.714	408	104	304	4900	10	190	4900	24	82.7	10	N.D.	14	N.D.	N.D.	N.D.	190
AA-P-1	2293284.955	705504.714	408	94	314	4.8	N.D.	N.D.	4.8	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.

APPENDIX A
TABLE A-1 EVS GROUNDWATER DATA SET
FOR FIGURES 2-1 THROUGH 2-7, 2-8, AND B-1 THROUGH B-33

Point ID	Easting	Northing	Surface Elevation (ft MSL)	Depth (ft bgs)	Depth Elevation (ft MSL)	MCB Concentration (ppm)	DCB Concentration (ppm)	Benzene Concentration (ppm)	Chlorobenzene Concentration (ppm)	Chloroaniline Concentration (ppm)	Total Chlorophenols Concentration (ppm)	Total Dichlorobenzene Concentration (ppm)	2,4-Dichlorophenol Concentration (ppm)	Phenol Concentration (ppm)	2,4,6-Trichlorophenol Concentration (ppm)	Total Methylphenols Concentration (ppm)	Acetone Concentration (ppm)	BTEX Concentration (ppm)
AA-P-1	2293284.955	705504.714	408	84	324	12	N.D.	N.D.	12	N.D.	5.9	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-1	2293284.955	705504.714	408	74	334	5.8	N.D.	N.D.	5.8	N.D.	6.4	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-1	2293284.955	705504.714	408	64	344	1	N.D.	N.D.	1	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	1.4	N.D.
AA-P-1	2293284.955	705504.714	408	54	354	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-1	2293284.955	705504.714	408	44	364	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-1	2293284.955	705504.714	408	34	374	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-1	2293284.955	705504.714	408	24	384	N.D.	N.D.	N.D.	N.D.	N.D.	0.21	N.D.	N.D.	N.D.	N.D.	N.D.	39	N.D.
AA-P-2	2292694.626	705577.77	410	122	288	480	N.D.	2800	480	2.4	4.6	N.D.	N.D.	120	N.D.	N.D.	N.A.	2800
AA-P-2	2292694.626	705577.77	410	114	296	500	1.5	6000	500	2.4	3.3	1.5	N.D.	180	N.D.	N.D.	N.A.	6000
AA-P-2	2292694.626	705577.77	410	104	306	490	N.D.	6900	490	N.D.	5.3	N.D.	N.D.	280	N.D.	N.D.	N.D.	7047
AA-P-2	2292694.626	705577.77	410	94	316	3.7	N.D.	N.D.	3.7	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-2	2292694.626	705577.77	410	84	326	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-2	2292694.626	705577.77	410	74	336	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-2	2292694.626	705577.77	410	64	346	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-2	2292694.626	705577.77	410	54	356	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-2	2292694.626	705577.77	410	44	366	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-2	2292694.626	705577.77	410	34	376	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-2	2292694.626	705577.77	410	24	386	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.86
AA-P-3	2291919.938	706378.705	410.5	125.5	285	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	16	N.D.
AA-P-3	2291919.938	706378.705	410.5	121.5	289	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
AA-P-3	2291919.938	706378.705	410.5	111.5	299	6.1	N.D.	1.3	6.1	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	1.77
AA-P-3	2291919.938	706378.705	410.5	101.5	309	0.45	N.D.	N.D.	0.45	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.54
AA-P-3	2291919.938	706378.705	410.5	91.5	319	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-3	2291919.938	706378.705	410.5	81.5	329	N.D.	N.D.	0.54	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.54
AA-P-3	2291919.938	706378.705	410.5	71.5	339	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.85
AA-P-3	2291919.938	706378.705	410.5	61.5	349	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.9
AA-P-3	2291919.938	706378.705	410.5	51.5	359	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-3	2291919.938	706378.705	410.5	41.5	369	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-P-3	2291919.938	706378.705	410.5	31.5	379	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
AA-Q-1	2290064.418	702408.594	420	127	293	1100	459	33	1100	3200	N.D.	459	N.D.	N.D.	N.D.	N.D.	N.D.	63.4
AA-Q-1	2290064.418	702408.594	420	120	300	310	13.5	28	310	27	10	13.5	N.D.	3.4	N.D.	N.D.	N.D.	30.1
AA-Q-1	2290064.418	702408.594	420	110	310	130	6.2	10	130	1.6	3.6	6.2	N.D.	N.D.	N.D.	N.D.	N.D.	13.28
AA-Q-1	2290064.418	702408.594	420	100	320	200	7.8	N.D.	200	5.2	4.6	7.8	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-Q-1	2290064.418	702408.594	420	90	330	220	12	1.4	220	110	3.7	12	N.D.	N.D.	N.D.	N.D.	N.D.	1.4
AA-Q-1	2290064.418	702408.594	420	80	340	130	17.6	N.D.	130	55	3.4	17.6	1	N.D.	N.D.	N.D.	N.D.	N.D.
AA-Q-1	2290064.418	702408.594	420	70	350	100	13.6	N.D.	100	14	1.6	13.6	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-Q-1	2290064.418	702408.594	420	60	360	240	29.5	N.D.	240	80	3	29.5	N.D.	N.D.	N.D.	N.D.	16	N.D.
AA-Q-1	2290064.418	702408.594	420	50	370	130	43.5	2.4	130	130	4.2	43.5	1	N.D.	N.D.	N.D.	11	3.4
AA-Q-2	2289414.428	701630.598	418	130	288	9.3	N.D.	0.35	9.3	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.35
AA-Q-2	2289414.428	701630.598	418	120	298	8.7	6.2	0.19	8.7	N.D.	N.D.	6.2	N.D.	N.D.	N.D.	N.D.	N.A.	0.94
AA-Q-2	2289414.428	701630.598	418	110	308	2.8	1.5	0.26	2.8	N.D.	N.D.	1.5	N.D.	N.D.	N.D.	N.D.	N.A.	0.83
AA-Q-2	2289414.428	701630.598	418	100	318	11	7.7	N.D.	11	N.D.	6	7.7	4.9	N.D.	1.1	N.D.	N.D.	N.D.
AA-Q-2	2289414.428	701630.598	418	90	328	15	8.8	N.D.	15	3.9	6.8	8.8	5.6	5.2	1.2	N.D.	N.D.	0.54
AA-Q-2	2289414.428	701630.598	418	80	338	16	6.8	N.D.	16	N.D.	70	6.8	46	N.D.	19	N.D.	N.D.	N.D.
AA-Q-2	2289414.428	701630.598	418	70	348	13	4.6	N.D.	13	N.D.	7.8	4.6	7.8	N.D.	N.D.	N.D.	N.D.	0.34
AA-Q-2	2289414.428	701630.598	418	60	358	11	3.3	N.D.	11	N.D.	50.8	3.3	43	14	N.D.	N.D.	N.D.	N.D.
AA-Q-3	2289105.417	701005.6	419	120	299	2.2	1.5	N.D.	2.2	N.D.	N.D.	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-Q-3	2289105.417	701005.6	419	110	309	1.6	N.D.	N.D.	1.6	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-Q-3	2289105.417	701005.6	419	100	319	2.5	1.4	N.D.	2.5	N.D.	N.D.	1.4	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-Q-3	2289105.417	701005.6	419	90	329	2.3	1.7	N.D.	2.3	N.D.	N.D.	1.7	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-Q-3	2289105.417	701005.6	419	80	339	13	4.6	N.D.	13	8.3	7.18	4.6	5.4	N.D.	1.8	N.D.	N.A.	N.D.
AA-Q-3	2289105.417	701005.6	419	70	349	8.4	3.6	N.D.	8.4	4.1	5.1	3.6	3.8	N.D.	1.3	N.D.	N.A.	0.36
AA-Q-3	2289105.417	701005.6	419	60	359	51	2.6	N.D.	51	5.8	28.6	2.6	19	N.D.	6	N.D.	N.A.	N.D.
AA-Q-3	2289105.417	701005.6	419	50	369	170	2.7	N.D.	170	N.D.	12.6	2.7	6.8	10	1.7	N.D.	N.D.	N.D.
AA-Q-4	2288510.417	700171.599	419	110	309	1.3	N.D.	N.D.	1.3	N.D.	0.49	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.

APPENDIX A
TABLE A-1 EVS GROUNDWATER DATA SET
FOR FIGURES 2-1 THROUGH 2-7, 2-8, AND B-1 THROUGH B-33

Point ID	Easting	Northing	Surface Elevation (ft MSL)	Depth (ft bgs)	Depth Elevation (ft MSL)	MCB Concentration (ppm)	DCB Concentration (ppm)	Benzene Concentration (ppm)	Chlorobenzene Concentration (ppm)	Chloroaniline Concentration (ppm)	Total Chlorophenols Concentration (ppm)	Total Dichlorobenzene Concentration (ppm)	2,4-Dichlorophenol Concentration (ppm)	Phenol Concentration (ppm)	2,4,6-Trichlorophenol Concentration (ppm)	Total Methylphenols Concentration (ppm)	Acetone Concentration (ppm)	BTEX Concentration (ppm)
AA-Q-4	2288510.417	700171.599	419	100	319	7.7	N.D.	0.18	7.7	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.18
AA-Q-4	2288510.417	700171.599	419	90	329	52	N.D.	N.D.	52	N.A.	4.2	N.D.	2.4	N.D.	N.D.	N.D.	N.D.	N.D.
AA-Q-4	2288510.417	700171.599	419	80	339	45	N.D.	0.38	45	N.A.	2.51	N.D.	2.1	N.D.	N.D.	N.D.	N.D.	1.6
AA-Q-4	2288510.417	700171.599	419	70	349	5.6	N.D.	N.D.	5.6	N.A.	2	N.D.	2	N.D.	N.D.	N.D.	N.D.	0.38
AA-Q-4	2288510.417	700171.599	419	60	359	9.8	N.D.	N.D.	9.8	N.A.	2.2	N.D.	2.2	N.D.	N.D.	N.D.	N.D.	1.16
AA-Q-4	2288510.417	700171.599	419	50	369	59	N.D.	N.D.	59	N.D.	7.6	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	0.36
AA-R-1	2290570	703255	420	131	289	2900	N.D.	120	2900	15000	N.D.	N.D.	N.D.	230	N.D.	N.D.	310	154
AA-R-1	2290570	703255	420	128	292	2000	55	100	2000	10000	55	55	N.D.	1100	N.D.	74	960	182
AA-R-1	2290570	703255	420	118	302	1700	260	200	1700	16000	140	260	N.D.	4500	N.D.	320	8200	590
AA-R-1	2290570	703255	420	108	312	3600	1338	240	3600	11000	540	1338	240	11000	N.D.	432	12000	2610
AA-R-1	2290570	703255	420	98	322	1000	1240	30	1000	4800	123	1240	98	130	N.D.	9.1	150	876
AA-R-1	2290570	703255	420	88	332	1200	198	120	1200	1300	22	198	N.D.	350	N.D.	22	470	168
AA-R-1	2290570	703255	420	78	342	4300	490	450	4300	25000	0.9	490	N.D.	1900	N.D.	200	2500	1420
AA-R-1	2290570	703255	420	68	352	7500	660	830	7500	51000	530	660	N.D.	26000	N.D.	2100	25000	4800
AA-R-1	2290570	703255	420	58	362	6900	370	800	6900	46000	1700	370	N.D.	27000	N.D.	1700	21000	4340
AA-R-1	2290570	703255	420	48	372	13000	270	1500	13000	40000	1700	270	N.D.	70000	N.D.	2997	83000	7260
AA-R-1	2290570	703255	420	28	392	2000	N.D.	510	2000	11000	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	579
AA-S-1	2291298.124	701787.942	411	124	287	78	N.D.	N.D.	78	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	0.79
AA-S-1	2291298.124	701787.942	411	114	297	410	N.D.	N.D.	410	N.D.	7.1	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
AA-S-1	2291298.124	701787.942	411	104	307	70	N.D.	N.D.	70	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
AA-S-1	2291298.124	701787.942	411	94	317	27	N.D.	N.D.	27	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-1	2291298.124	701787.942	411	84	327	22	N.D.	N.D.	22	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.46
AA-S-1	2291298.124	701787.942	411	74	337	4.8	N.D.	N.D.	4.8	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-1	2291298.124	701787.942	411	64	347	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
AA-S-1	2291298.124	701787.942	411	54	357	0.44	N.D.	N.D.	0.44	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-1	2291298.124	701787.942	411	44	367	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-1	2291298.124	701787.942	411	34	377	N.D.	1.4	N.D.	N.D.	N.D.	N.D.	1.4	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-1	2291298.124	701787.942	411	24	387	N.D.	1.5	3	N.D.	N.D.	N.D.	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	3.41
AA-S-2	2291199.56	701885.681	412	118.5	293.5	290	N.D.	N.D.	290	N.D.	5.9	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-2	2291199.56	701885.681	412	118	294	340	N.D.	N.D.	340	N.D.	10	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-2	2291199.56	701885.681	412	108	304	140	N.D.	N.D.	140	N.D.	4.4	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-2	2291199.56	701885.681	412	98	314	35	N.D.	N.D.	35	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-2	2291199.56	701885.681	412	88	324	24	N.D.	N.D.	24	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-2	2291199.56	701885.681	412	78	334	8.9	N.D.	N.D.	8.9	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-2	2291199.56	701885.681	412	68	344	1.6	N.D.	N.D.	1.6	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-2	2291199.56	701885.681	412	58	354	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-2	2291199.56	701885.681	412	48	364	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	0.57
AA-S-2	2291199.56	701885.681	412	38	374	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-2	2291199.56	701885.681	412	28	384	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-3	2291066.539	701927.892	416	132	284	250	N.D.	N.D.	250	1.7	4.3	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	1.2
AA-S-3	2291066.539	701927.892	416	124	292	460	2.8	N.D.	460	2.4	7.9	2.8	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-3	2291066.539	701927.892	416	114	302	530	N.D.	N.D.	530	3.7	10	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	1.4
AA-S-3	2291066.539	701927.892	416	104	312	180	1.3	N.D.	180	N.D.	4.2	1.3	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-3	2291066.539	701927.892	416	94	322	51	N.D.	N.D.	51	N.D.	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-3	2291066.539	701927.892	416	84	332	16	N.D.	N.D.	16	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-3	2291066.539	701927.892	416	74	342	3.7	N.D.	N.D.	3.7	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-3	2291066.539	701927.892	416	64	352	2.1	1.4	N.D.	2.1	N.D.	N.D.	1.4	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-3	2291066.539	701927.892	416	54	362	1.4	N.D.	N.D.	1.4	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
AA-S-3	2291066.539	701927.892	416	44	372	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.34
AA-S-3	2291066.539	701927.892	416	34	382	1.2	N.D.	N.D.	1.2	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
AA-S-3	2291066.539	701927.892	416	24	392	1.8	N.D.	0.18	1.8	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	0.59
B-24A	2290763	703439.6	419.5	23	395.5	840	N.D.	710	840	N.D.	223000	N.D.	35000	300000	38000	5000	48000	710
B-24C	2290762.9	703439.6	421	62	359	7300	N.D.	1900	7300	15000	132500	N.D.	73000	64000	9500	6200	2300	4020
B-25A	2291280	703311	426	30.5	395.5	7900	N.D.	N.A.	7900	N.D.	228000	N.D.	120000	290000	12000	60000	9000	N.D.
B-25B	2291279.9	703311	425	42.5	382.5	14000	N.D.	N.A.	14000	N.D.	111000	N.D.	63000	340000	N.D.	N.D.	N.A.	510

APPENDIX A
TABLE A-1 EVS GROUNDWATER DATA SET
FOR FIGURES 2-1 THROUGH 2-7, 2-8, AND B-1 THROUGH B-33

Point ID	Easting	Northing	Surface Elevation (ft MSL)	Depth (ft bgs)	Depth Elevation (ft MSL)	MCB Concentration (ppm)	DCB Concentration (ppm)	Benzene Concentration (ppm)	Chlorobenzene Concentration (ppm)	Chloroaniline Concentration (ppm)	Total Chlorophenols Concentration (ppm)	Total Dichlorobenzene Concentration (ppm)	2,4-Dichlorophenol Concentration (ppm)	Phenol Concentration (ppm)	2,4,6-Trichlorophenol Concentration (ppm)	Total Methylphenols Concentration (ppm)	Acetone Concentration (ppm)	BTEX Concentration (ppm)
B-28A	2290369	702607.9	421	28	393	160	N.D.	N.D.	160	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
B-28B	2290368.9	702607.9	421	42.5	378.5	3300	5500	N.A.	3300	92000	81200	5500	41000	77000	13000	22000	N.A.	970
B-29A	2290846	702438	427	28.5	395.5	590	N.D.	N.A.	590	N.D.	235000	N.D.	32000	320000	13000	256000	N.A.	300
B-29B	2290845.9	702438	427	42.5	384.5	1200	N.D.	N.D.	1200	N.D.	250000	N.D.	61000	680000	9000	160000	N.D.	320
GM-17A	2294280	702632.7	410	28	382	220000	N.D.	760000	220000	N.D.	59	N.D.	N.D.	100	N.D.	N.D.	N.D.	760000
GM-17B	2294279.9	702632.7	410	68	342	3300	145	N.D.	3300	N.D.	17	145	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
GM-17C	2294280.1	702632.7	409	97	312	1500	264	N.D.	1500	24	N.D.	264	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
GM-19A	2292081	702222.4	410.5	22.5	388	7.2	N.D.	N.D.	7.2	N.D.	256	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
GM-19B	2292081	702222.4			339.5	N.A.	N.A.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
GM-19C	2292080.9	702222.4	410.5	70.5	340	1400	480	16	1400	N.D.	37	480	15	N.D.	N.D.	N.D.	N.D.	16
GM-20A	2292755	702839.1	410.5	15	395.5	140	28	200	140	100	59	28	17	N.D.	28	N.D.	N.D.	242
GM-20B	2292754.9	702839.1	410.5	86	324.5	5200	1200	N.D.	5200	63	32	1200	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
GM-4A	2294448	703356.5	403	20.5	382.5	46	N.D.	N.D.	46	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
GM-4B	2294447.9	703356.5	404	77	327	1600	N.D.	2800	1600	44	10	N.D.	N.D.	64	N.D.	N.D.	N.D.	2800
GM-4C	2294448.1	703356.5	404.5	97.5	307	9600	N.D.	19000	9600	46	22	N.D.	N.D.	170	N.D.	N.D.	N.D.	19000
GM-6A	2294116	702030	413	21.5	391.5	30000	50	3600	30000	N.D.	78	50	N.D.	30	N.D.	N.D.	N.D.	3600
GM-6B	2294115.9	702030	413	78	335	18000	217	N.D.	18000	N.D.	62	217	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
GM-7	2293251	701891.3	412.5	28.5	384	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
K-1	2297248.7	702637.3	413.07	113.25	299.82	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
K-3	2296185.9	702591.7	412.13	109.58	302.55	1600	2740	N.D.	1600	3.5	32	2740	20	N.D.	N.D.	4.6	N.D.	N.D.
K-4	2296048.7	702975.9	409.5	110	299.5	15000	3160	150	15000	380	169.3	3160	8.3	N.D.	N.D.	N.D.	N.D.	150
K-5	2295917.6	702200.9	412.27	109.5	302.77	4500	132	N.D.	4500	4.6	31.0015	132	N.D.5	N.D.	N.D.	N.D.	N.D.	N.D.
K-6	2294900.8	701842.4	410.5	110	300.5	160	878	16	160	89	N.D.	878	N.D.	N.D.	N.D.	N.D.	N.D.	20.9
K-7	2294707.9	701947.3	408.5	108.5	300	520	749	30	520	33	N.D.	749	N.D.	N.D.	N.D.	N.D.	N.D.	30
K-8	2294328.4	702026.8	408.5	107.5	301	440	297	26	440	19	5.4	297	3.1	N.D.	N.D.	26	N.D.	26
K-9	2294395.8	702985.5	403.7	103	300.7	2600	160	N.D.	2600	N.D.9	3.1	160	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
MW-7B	2293328	702124.6	411	70	341	6300	1030	N.D.	6300	120	60	1030	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
MW-7C	2293327.9	702124.6	411.05	95	316.05	10000	1005	N.D.	10000	130	41	1005	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA1-GPA	2298273	704724.7	411	77	334	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
TRA1-GPA	2298273	704724.7	411	43	368	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
TRA1-GPA	2298273	704724.7	411	23	388	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
TRA1-GPB	2297411	702640.2	413	73	340	N.D.	N.D.	7800	N.D.	N.D.	N.D.	N.D.	N.D.	26	N.D.	N.D.	N.D.	8950
TRA1-GPB	2297411	702640.2	413	43	370	N.D.	N.D.	37000	N.D.	N.D.	N.D.	N.D.	N.D.	81	N.D.	N.D.	N.D.	44420
TRA1-GPB	2297411	702640.2	413	21	392	N.D.	N.D.	150	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	174
TRA1-GPC	2296720	700180.5	414	74	340	N.D.	N.D.	29	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	569
TRA1-GPC	2296720	700180.5	414	44	370	N.D.	N.D.	390	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	493
TRA1-GPC	2296720	700180.5	414	15	395.5	88	N.D.	N.D.	88	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPA	2297253	705272.2	410	70	340	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPA	2297253	705272.2	410	45	365	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPA	2297253	705272.2	410	25	385	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPB	2296811	703954.6	411	71	340	N.D.	N.D.	18	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	18
TRA2-GPB	2296811	703954.6	411	45	366	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPB	2296811	703954.6	411	25	386	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPC	2296445	703637	411	73	338	1200	N.D.	3200	1200	91	10	N.D.	N.D.	110	N.D.	N.D.	N.D.	3200
TRA2-GPC	2296445	703637	411	43	368	120	14	N.D.	120	N.D.	N.D.	14	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPC	2296445	703637	411	25	386	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPD	2296251	702967	411	72	339	1200	831	1300	1200	31	N.D.	831	N.D.	11	N.D.	33	N.D.	2279
TRA2-GPD	2296251	702967	411	41	370	1800	960	49	1800	33	N.D.	960	N.D.	N.D.	N.D.	N.D.	N.D.	49
TRA2-GPD	2296251	702967	411	21	390	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPE	2296079	702223.7	412	72	340	7.2	N.D.	N.D.	7.2	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPE	2296079	702223.7	412	42	370	540	N.D.	N.D.	540	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPE	2296079	702223.7	412	21	391	15	47	N.D.	15	N.D.	N.D.	47	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPF	2295946	701506.5	412	58	354	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPF	2295946	701506.5	412	42	370	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPF	2295946	701506.5	412	15	395.5	9.9	N.D.	N.D.	9.9	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.

APPENDIX A
TABLE A-1 EVS GROUNDWATER DATA SET
FOR FIGURES 2-1 THROUGH 2-7, 2-8, AND B-1 THROUGH B-33

Point ID	Easting	Northing	Surface Elevation (ft MSL)	Depth (ft bgs)	Depth Elevation (ft MSL)	MCB Concentration (ppm)	DCB Concentration (ppm)	Benzene Concentration (ppm)	Chlorobenzene Concentration (ppm)	Chloroaniline Concentration (ppm)	Total Chlorophenols Concentration (ppm)	Total Dichlorobenzene Concentration (ppm)	2,4-Dichlorophenol Concentration (ppm)	Phenol Concentration (ppm)	2,4,6-Trichlorophenol Concentration (ppm)	Total Methylphenols Concentration (ppm)	Acetone Concentration (ppm)	BTEX Concentration (ppm)
TRA2-GPG	2295909	700756.5	413	73	340	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPG	2295909	700756.5	413	43	370	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA2-GPG	2295909	700756.5	413	15	395.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPA	2294041	704995.7	406	105	301	850	40	N.D.	850	N.D.	N.D.	40	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPA	2294041	704995.7	406	66	340	10	N.D.	N.D.	10	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPA	2294041	704995.7	406	36	370	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPA	2294041	704995.7	406	13	393	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPB	2293918	704440	416	119	297	8300	53	N.D.	8300	22	24	53	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPB	2293918	704440	416	76	340	400	N.D.	N.D.	400	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPB	2293918	704440	416	49	367	8.7	N.D.	N.D.	8.7	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPB	2293918	704440	416	29	387	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPC	2293362	702688	413	119	294	3200	302	120	3200	N.D.	34	302	N.D.	N.D.	N.D.	N.D.	N.D.	120
TRA4-GPC	2293362	702688	413	73	340	57000	33	160000	57000	140	22	33	N.D.	200	N.D.	N.D.	N.D.	160000
TRA4-GPC	2293362	702688	413	43	370	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPC	2293362	702688	413	21	392	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPD	2292659	700504	418	119	299	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPD	2292659	700504	418	78	340	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPD	2292659	700504	418	48	370	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPD	2292659	700504	418	28	390	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPE	2292430	699880	419	114	305	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPE	2292430	699880	419	79	340	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPE	2292430	699880	419	49	370	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA4-GPE	2292430	699880	419	27	392	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA6-GPC	2290925	704344.1	425	121	304	410	N.D.	9.6	410	180	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	9.6
TRA6-GPC	2290925	704344.1	425	85	340	9300	140	1300	9300	37000	N.D.	140	N.D.	N.D.	N.D.	N.D.	N.D.	1453
TRA6-GPC	2290925	704344.1	425	65	360	6500	32	1100	6500	8600	N.D.	32	N.D.	N.D.	N.D.	N.D.	N.D.	1191
TRA6-GPC	2290925	704344.1	425	24	395.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA6-GPD	2289695	702017	420	114	306	6.3	N.D.	N.D.	6.3	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA6-GPD	2289695	702017	420	85	335	25	N.D.	N.D.	25	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
TRA6-GPD	2289695	702017	420	55	365	5.4	N.D.	N.D.	5.4	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
UAA-1/OS-1	2294186.408	705375.587	406	110	296	710	42.3	3.4	710	N.D.	6.4	42.3	N.D.	N.D.	N.D.	N.D.	N.A.	3.78
UAA-1/OS-1	2294186.408	705375.587	406	100	306	400	21.8	2.5	400	N.D.	1.7	21.8	N.D.	N.D.	N.D.	N.D.	N.A.	3.12
UAA-1/OS-1	2294186.408	705375.587	406	90	316	450	11.2	0.23	450	N.D.	5.4	11.2	N.D.	N.D.	N.D.	N.D.	N.A.	0.23
UAA-1/OS-1	2294186.408	705375.587	406	80	326	280	1.8	N.D.	280	N.D.	3.3	1.8	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
UAA-1/OS-1	2294186.408	705375.587	406	70	336	2.4	N.D.	N.D.	2.4	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
UAA-1/OS-1	2294186.408	705375.587	406	60	346	2	N.D.	N.D.	2	N.D.	0.05	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
UAA-1/OS-1	2294186.408	705375.587	406	50	356	0.42	N.D.	N.D.	0.42	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
UAA-1/OS-1	2294186.408	705375.587	406	40	366	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
UAA-1/OS-1	2294186.408	705375.587	406	30	376	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
UAA-1/OS-1	2294186.408	705375.587	406	20	386	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
UAA-2/OS-2	2293195.407	701744.598	412	124	288	2000	449.4	24	2000	2.4	28.3	449.4	8.3	N.D.	N.D.	N.D.	N.D.	24
UAA-2/OS-2	2293195.407	701744.598	412	120	292	760	900	N.D.	760	2.8	21.6	900	12	N.D.	N.D.	N.D.	N.D.	N.D.
UAA-2/OS-2	2293195.407	701744.598	412	110	302	2500	1265	17	2500	9.7	48	1265	16	N.D.	N.D.	N.D.	N.D.	17
UAA-2/OS-2	2293195.407	701744.598	412	100	312	2600	379.8	20	2600	3.1	26	379.8	8	N.D.	N.D.	N.D.	N.D.	20
UAA-2/OS-2	2293195.407	701744.598	412	90	322	1800	263.3	13	1800	8.6	32.7	263.3	8.7	N.D.	N.D.	N.D.	N.D.	13
UAA-2/OS-2	2293195.407	701744.598	412	80	332	62	N.D.	55	62	6	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	55
UAA-2/OS-2	2293195.407	701744.598	412	70	342	81	N.D.	24	81	3.7	1.6	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	24
UAA-2/OS-2	2293195.407	701744.598	412	60	352	79	N.D.	15	79	4.4	2.4	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	15
UAA-2/OS-2	2293195.407	701744.598	412	50	362	52	N.D.	12	52	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	12
UAA-2/OS-2	2293195.407	701744.598	412	40	372	N.D.	N.D.	0.27	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	0.27
UAA-2/OS-2	2293195.407	701744.598	412	30	382	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
UAA-2/OS-2	2293195.407	701744.598	412	20	392	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
UAA-3/OS-3	2292594.413	701059.598	405	116	289	360	1529	1.8	360	9.8	64.2	1529	53	N.D.	1.5	N.D.	N.A.	1.8
UAA-3/OS-3	2292594.413	701059.598	405	114	291	570	1841	4.3	570	N.D.	77	1841	59	N.D.	1	N.D.	N.A.	4.3
UAA-3/OS-3	2292594.413	701059.598	405	104	301	2100	2253	24	2100	2100	73.8	2253	52	N.D.	7.8	N.D.	N.A.	24

APPENDIX A
TABLE A-1 EVS GROUNDWATER DATA SET
FOR FIGURES 2-1 THROUGH 2-7, 2-8, AND B-1 THROUGH B-33

Point ID	Easting	Northing	Surface Elevation (ft MSL)	Depth (ft bgs)	Depth Elevation (ft MSL)	MCB Concentration (ppm)	DCB Concentration (ppm)	Benzene Concentration (ppm)	Chlorobenzene Concentration (ppm)	Chloroaniline Concentration (ppm)	Total Chlorophenols Concentration (ppm)	Total Dichlorobenzene Concentration (ppm)	2,4-Dichlorophenol Concentration (ppm)	Phenol Concentration (ppm)	2,4,6-Trichlorophenol Concentration (ppm)	Total Methylphenols Concentration (ppm)	Acetone Concentration (ppm)	BTEX Concentration (ppm)
UAA-3/OS-3	2292594.413	701059.598	405	94	311	2100	1037	55	2100	800	18.9	1037	9.4	N.D.	N.D.	N.D.	N.A.	55
UAA-3/OS-3	2292594.413	701059.598	405	84	321	1300	175	63	1300	470	16.2	175	2.2	N.D.	N.D.	N.D.	N.A.	63
UAA-3/OS-3	2292594.413	701059.598	405	74	331	150	N.D.	7.4	150	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	7.7
UAA-3/OS-3	2292594.413	701059.598	405	64	341	31	N.D.	0.14	31	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	0.14
UAA-3/OS-3	2292594.413	701059.598	405	54	351	160	N.D.	1.2	160	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	1.2
UAA-3/OS-3	2292594.413	701059.598	405	44	361	160	N.D.	3.6	160	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	3.6
UAA-3/OS-3	2292594.413	701059.598	405	34	371	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.
UAA-3/OS-3	2292594.413	701059.598	405	24	381	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.A.	N.D.

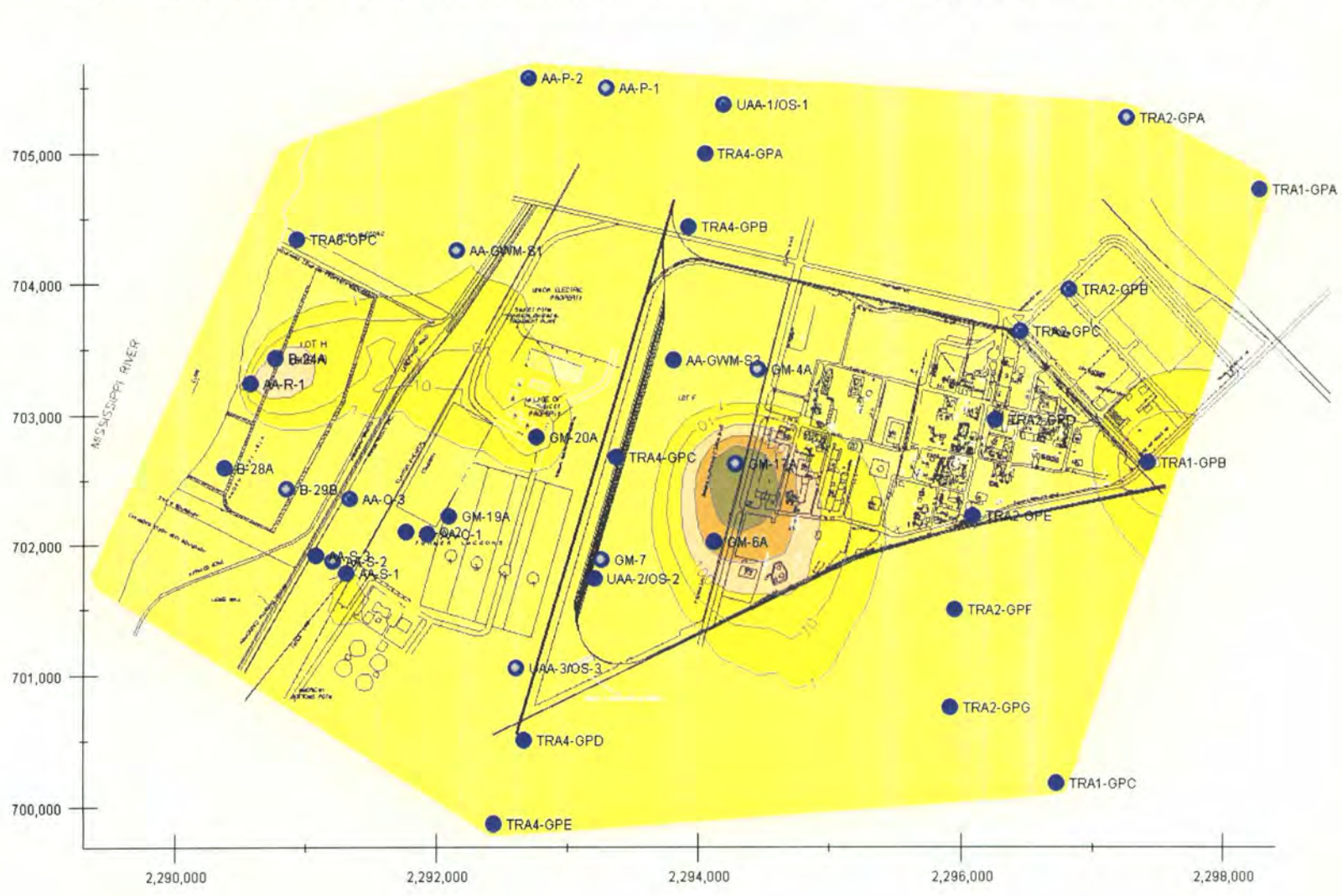
Notes:
1) N.D. means the sample was non-detect for the analyte.
2) N.A. means the sample was not analyzed for the analyte.

Groundwater Isoconcentration Maps

Plan View

Maximum Concentration of Benzene in Groundwater – SHU (Elevation 395 to 380)

URS



<1 ug/l

1 to 10 ug/l

10 to 100 ug/l

100 to 1000 ug/l

1000 to 10000 ug/l

>10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: B-1

Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

Maximum Concentration of Benzene in Groundwater – MHU (Elevation 380 to 350)

URS

<1 ug/l
1 to 10 ug/l
10 to 100 ug/l
100 to 1000 ug/l
1000 to 10000 ug/l
>10000 ug/l

SOLUTIA

Date: 07/14/2005

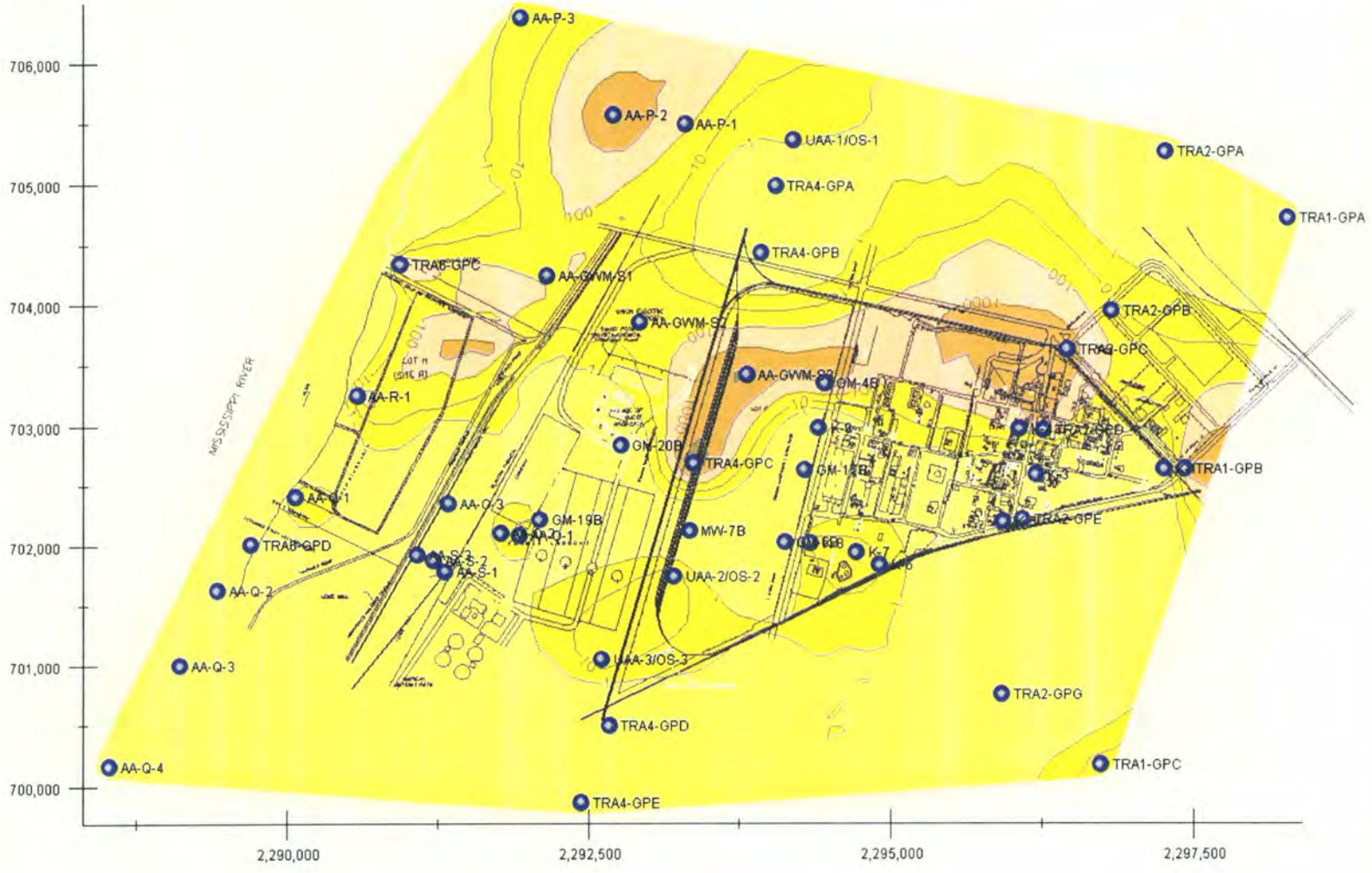
Figure Number: B-2

Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View Maximum Concentration of Benzene in Groundwater – DHU (Elevation 350 to Bedrock)

URS



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005

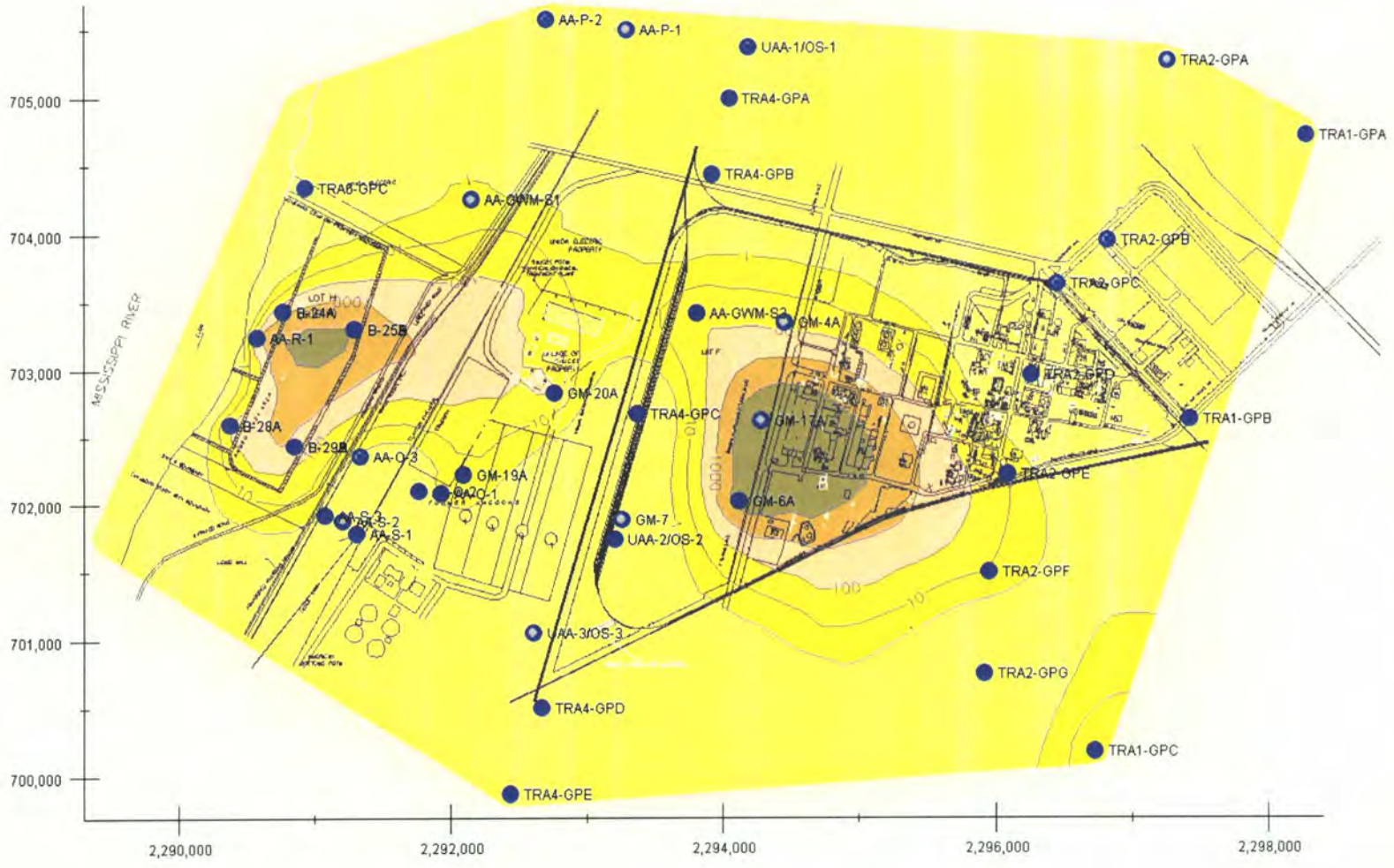
Figure Number: B-3

Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View Maximum Concentration of Chlorobenzene in Groundwater – SHU (Elevation 395 to 380)

URS



<1 ug/l

1 to 10 ug/l

10 to 100 ug/l

100 to 1000 ug/l

1000 to 10000 ug/l

>10000 ug/l

SOLUTIA

Date: 07/14/2005

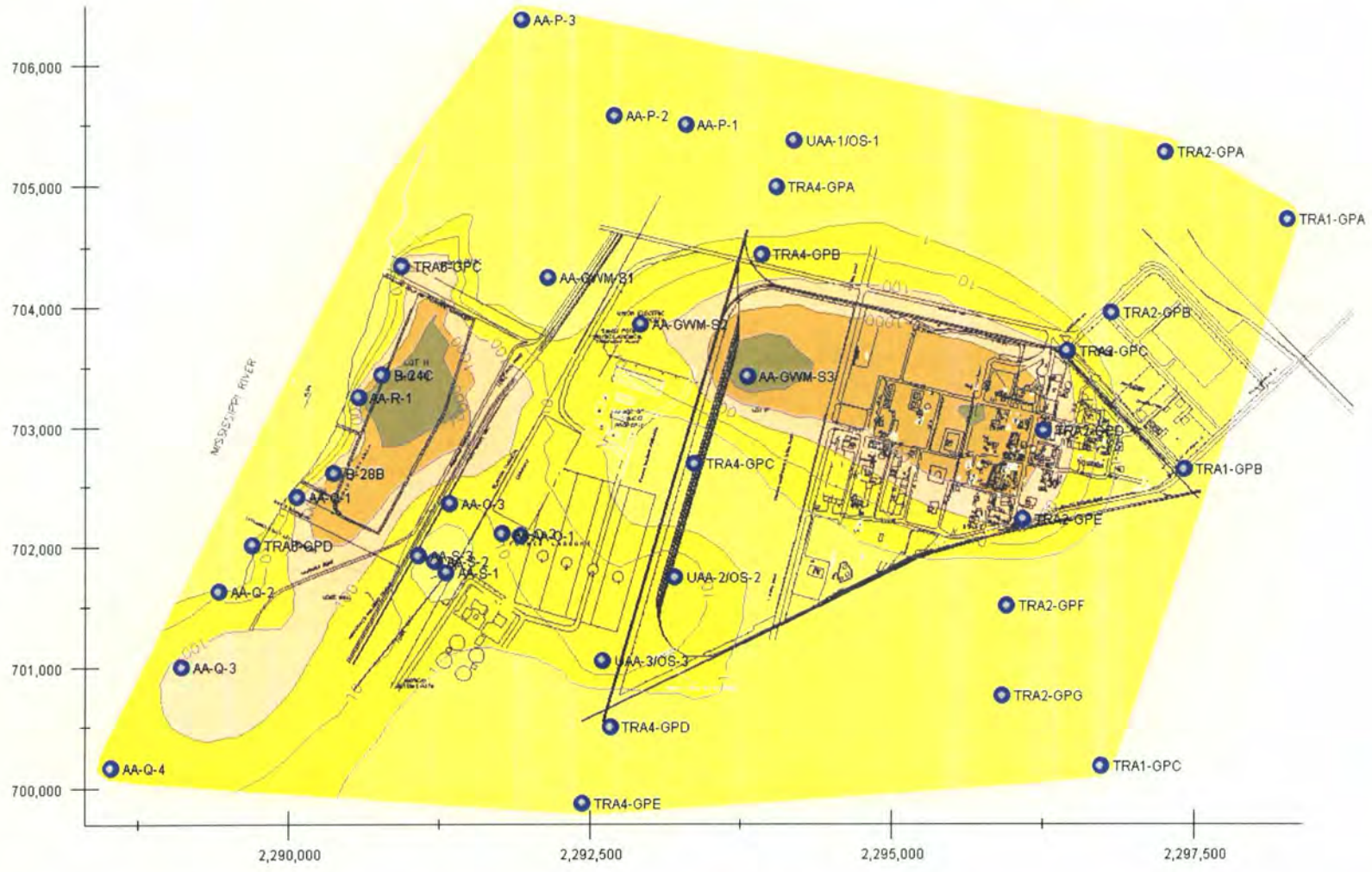
Figure Number: B-4

Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View Maximum Concentration of Chlorobenzene in Groundwater – MHU (Elevation 380 to 350)

URS



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

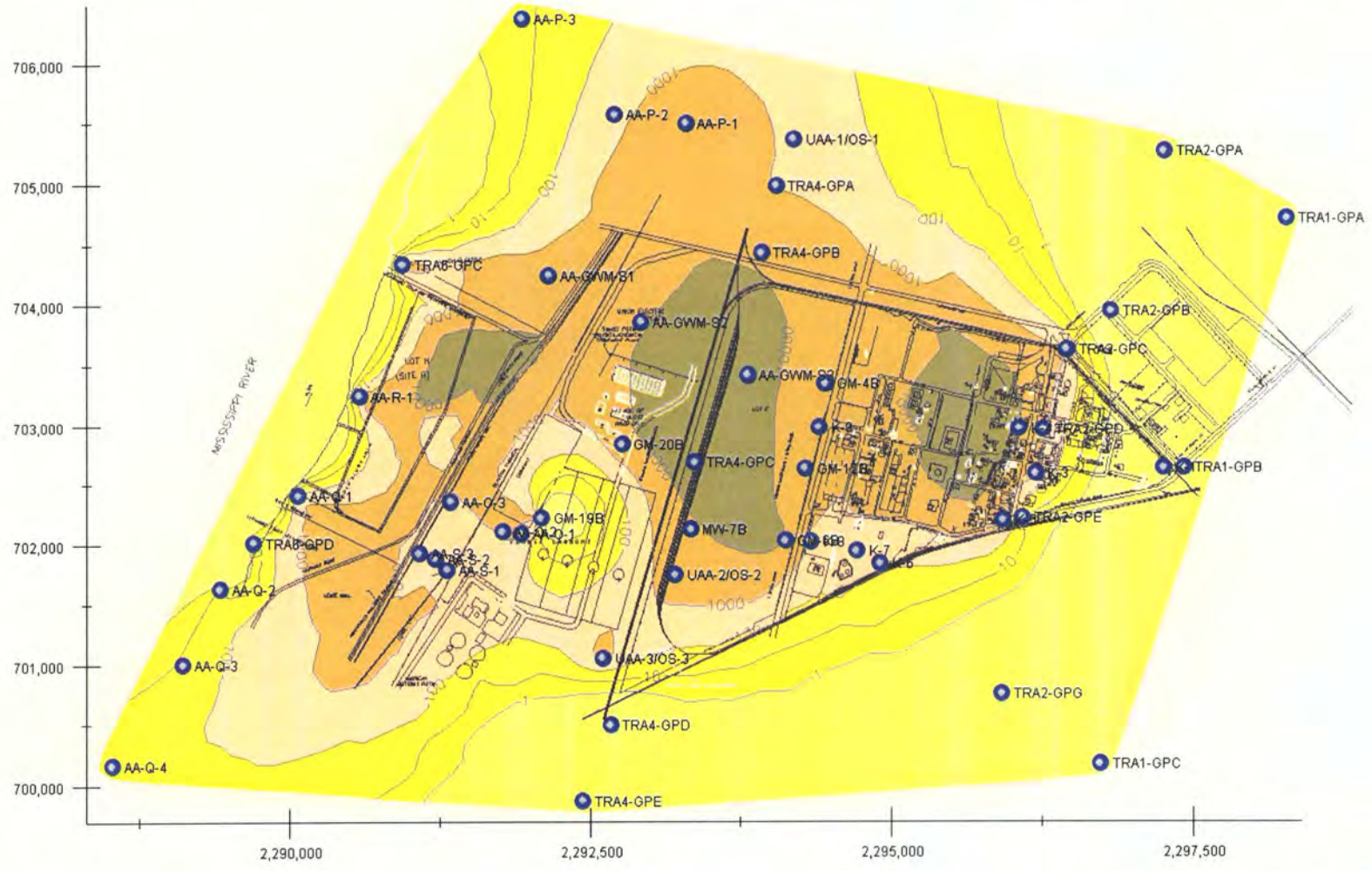
Date: 07/14/2005	
Figure Number: B-5	
Project Number: 21561601.00000	

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of Chlorobenzene in Groundwater – DHU (Elevation 350 to Bedrock)



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

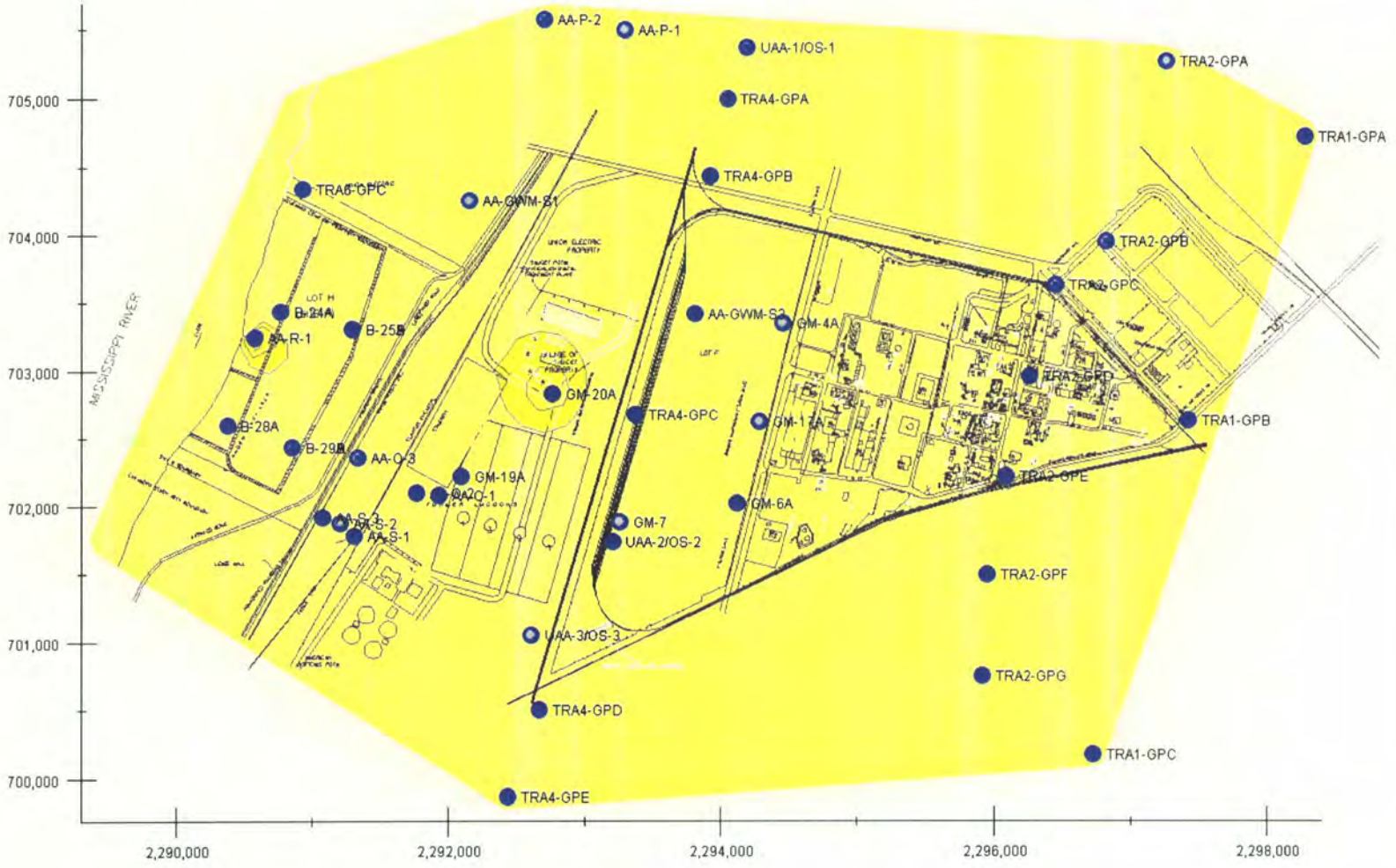
SOLUTIA

Date: 07/14/2005	
Figure Number: B-6	
Project Number: 21561601.00000	

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View Maximum Concentration of Chloroanilines in Groundwater – SHU (Elevation 395 to 380)

URS



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005	
Figure Number: B-7	
Project Number: 21561601.00000	

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of Chloroanilines in Groundwater – MHU (Elevation 380 to 350)

<1 ug/l
1 to 10 ug/l
10 to 100 ug/l
100 to 1000 ug/l
1000 to 10000 ug/l
>10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: B-8

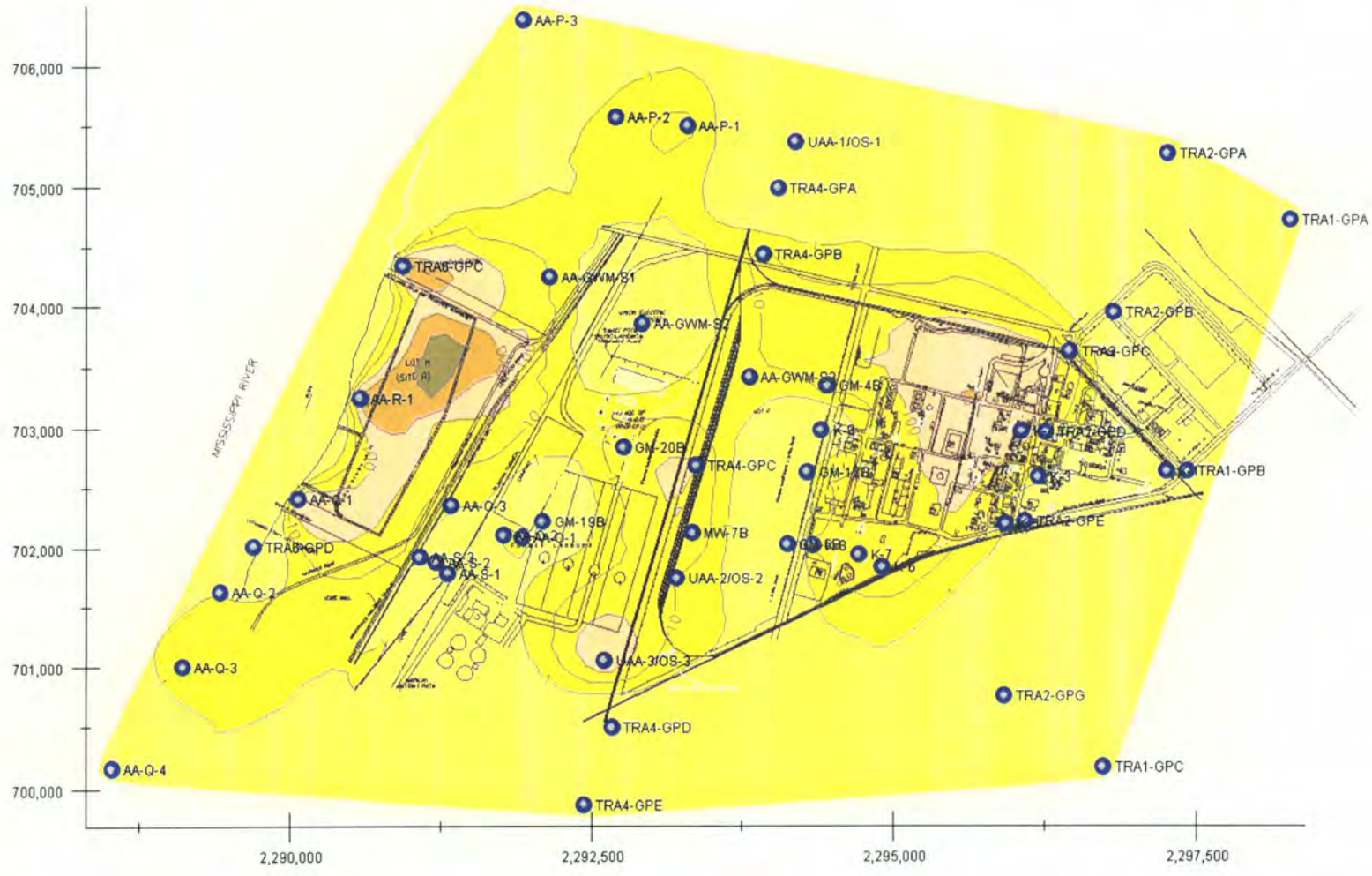
Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of Chloroanilines in Groundwater – DHU (Elevation 350 to Bedrock)



- <1 ug/l
- 1 to 10 ug/l
- 10 to 100 ug/l
- 100 to 1000 ug/l
- 1000 to 10000 ug/l
- >10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: B-9

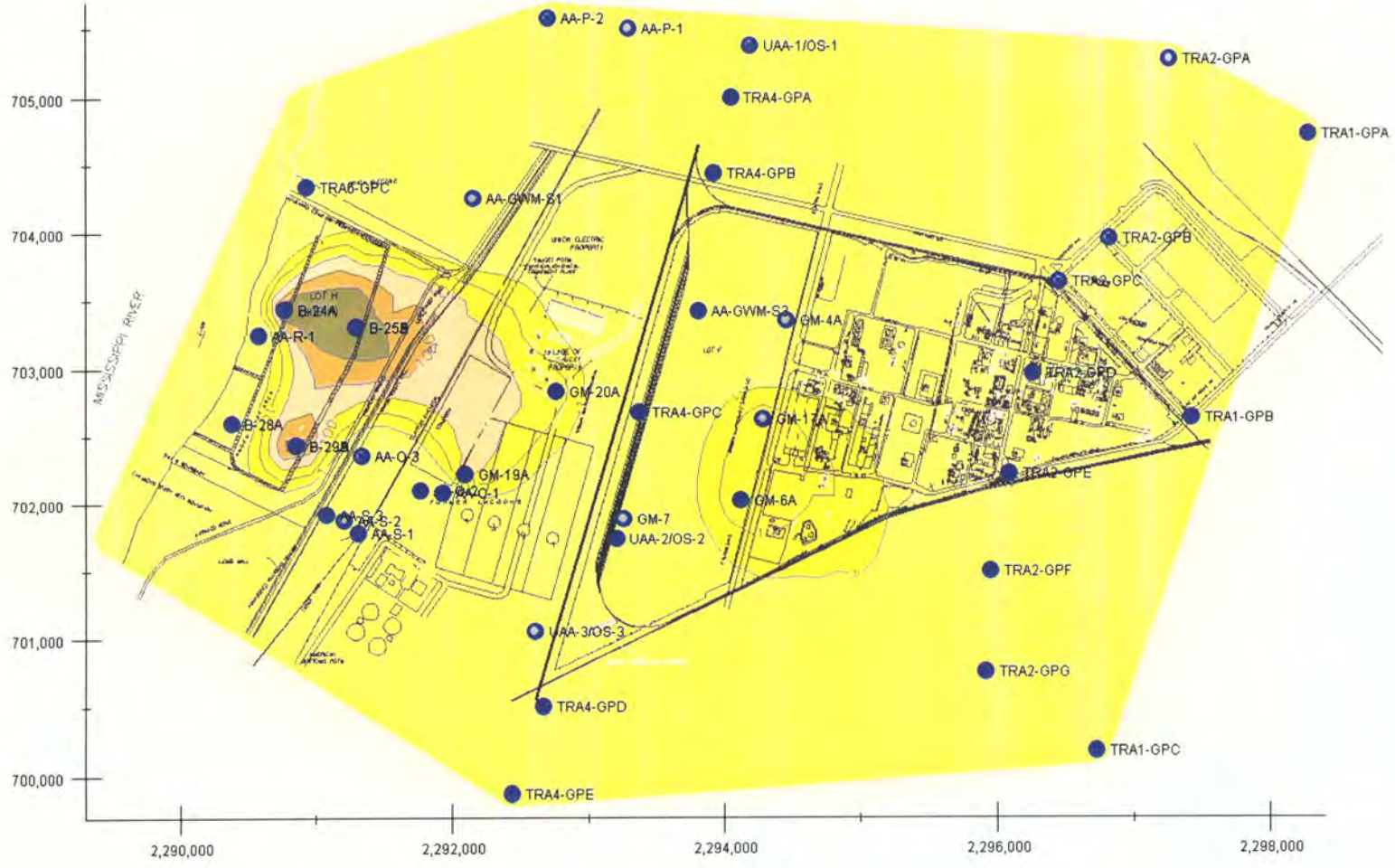
Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of Chlorophenols in Groundwater – SHU (Elevation 395 to 380)



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005	
Figure Number:	B-10
Project Number:	21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of Chlorophenols in Groundwater – MHU (Elevation 380 to 350)

<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

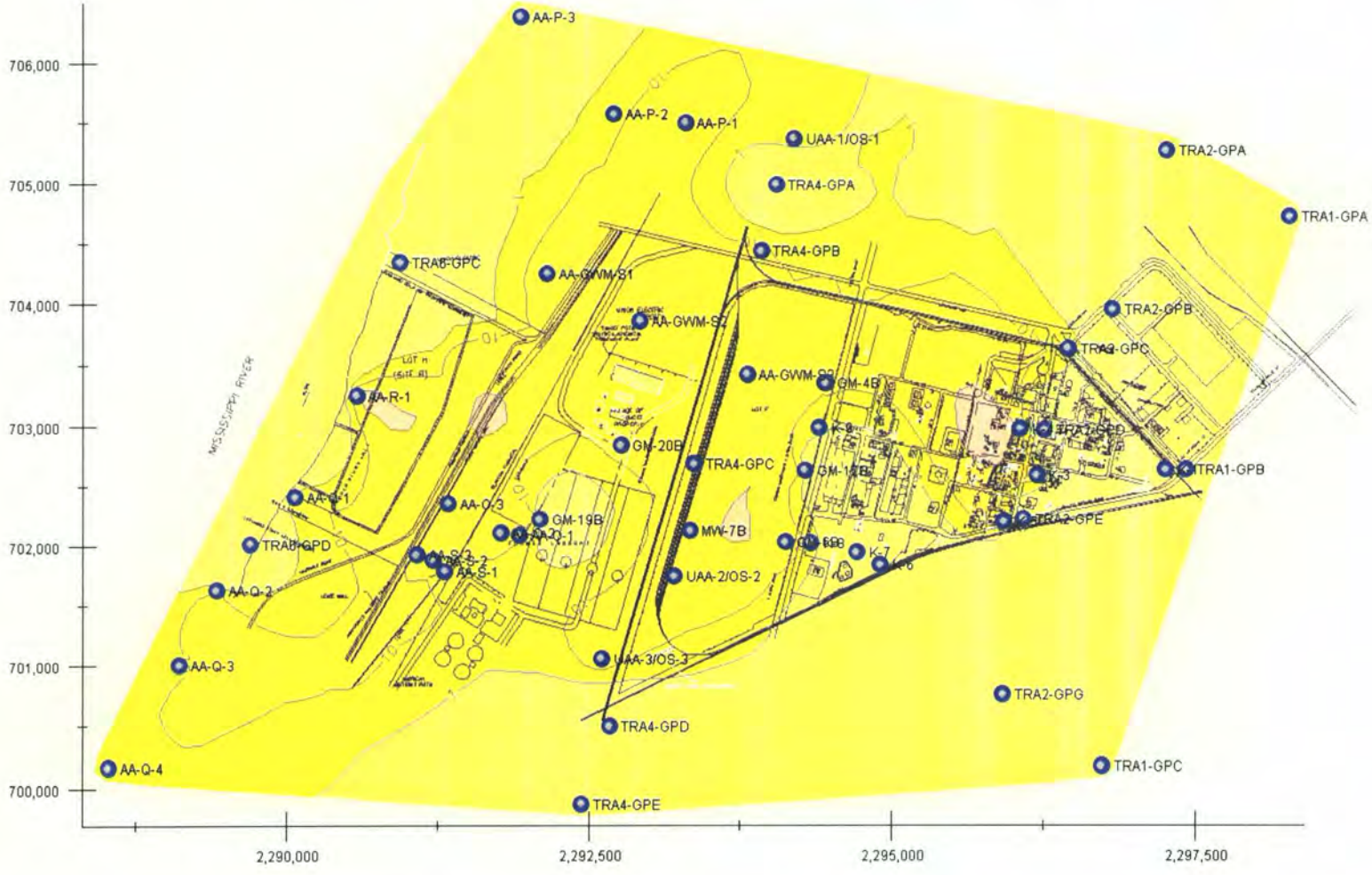
Date: 07/14/2005	
Figure Number:	B-11
Project Number:	21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of Chlorophenols in Groundwater – DHU (Elevation 350 to Bedrock)



<1 ug/l

1 to 10 ug/l

10 to 100 ug/l

100 to 1000 ug/l

1000 to 10000 ug/l

>10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: B-12

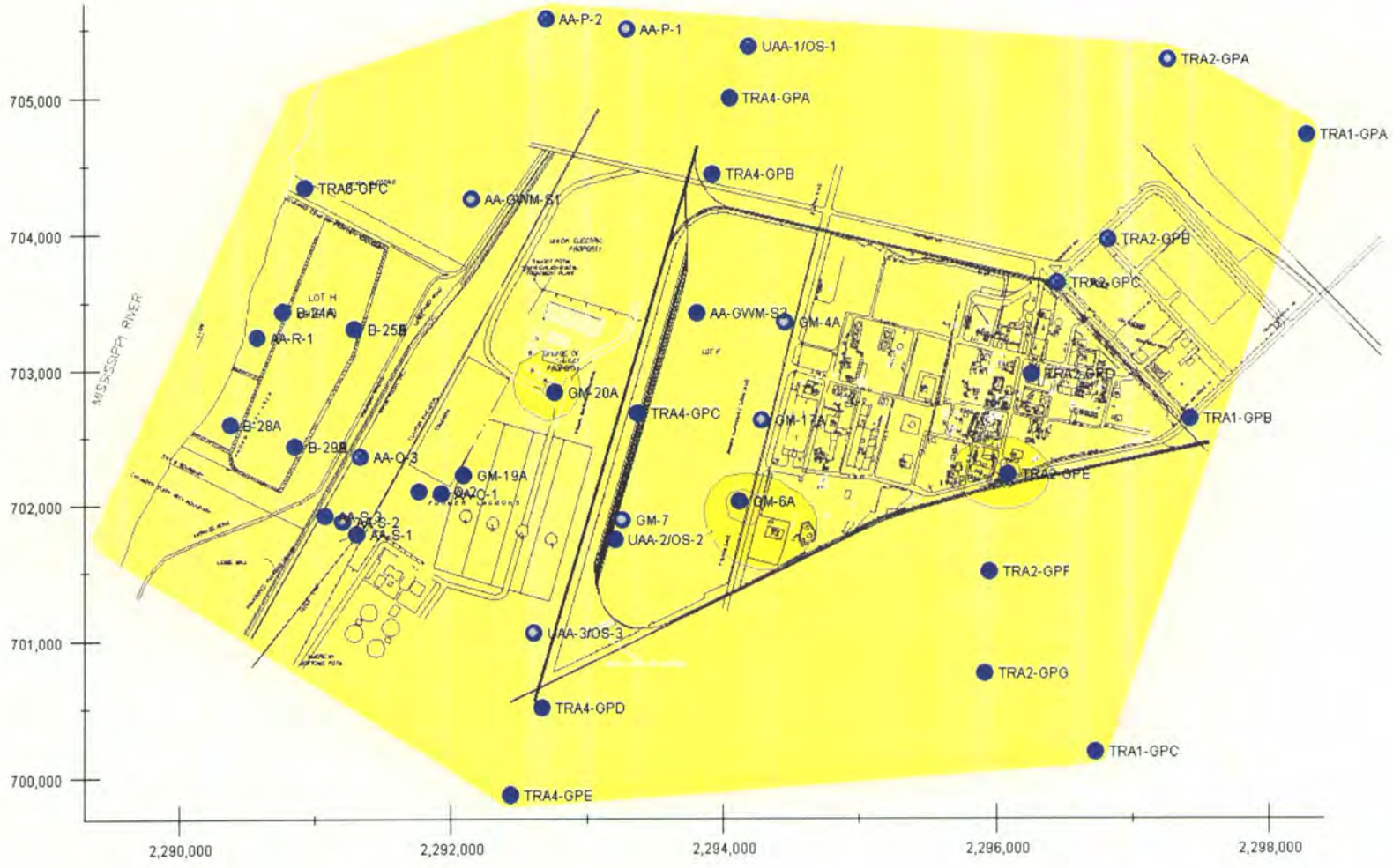
Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of Dichlorobenzenes in Groundwater – SHU (Elevation 395 to 380)



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

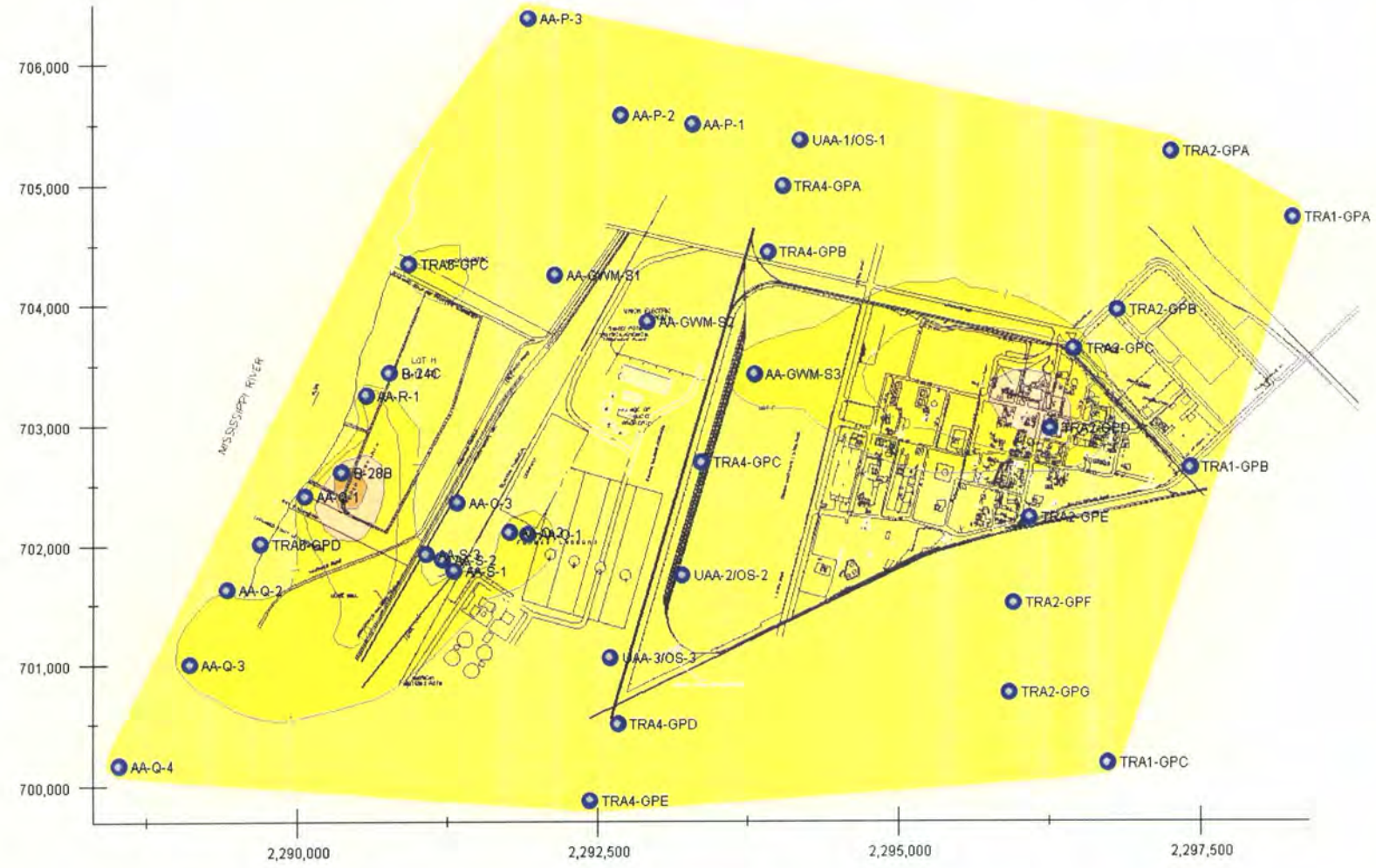
Date: 07/14/2005	
Figure Number: B-13	
Project Number: 21561601.00000	

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of Dichlorobenzenes in Groundwater – MHU (Elevation 380 to 350)



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

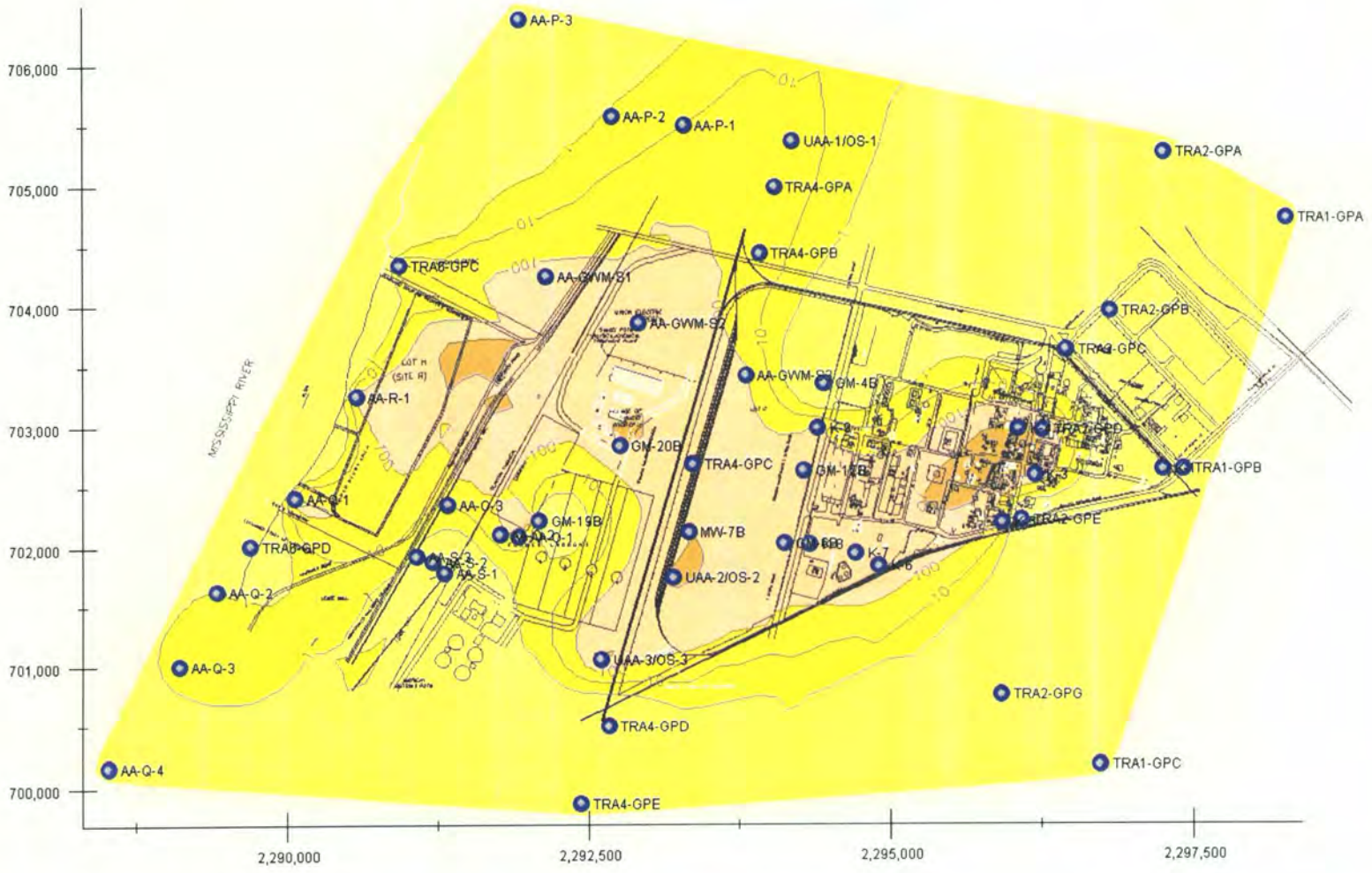
Date: 07/14/2005	
Figure Number: B-14	
Project Number: 21561601.00000	

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of Dichlorobenzenes in Groundwater – DHU (Elevation 350 to Bedrock)



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

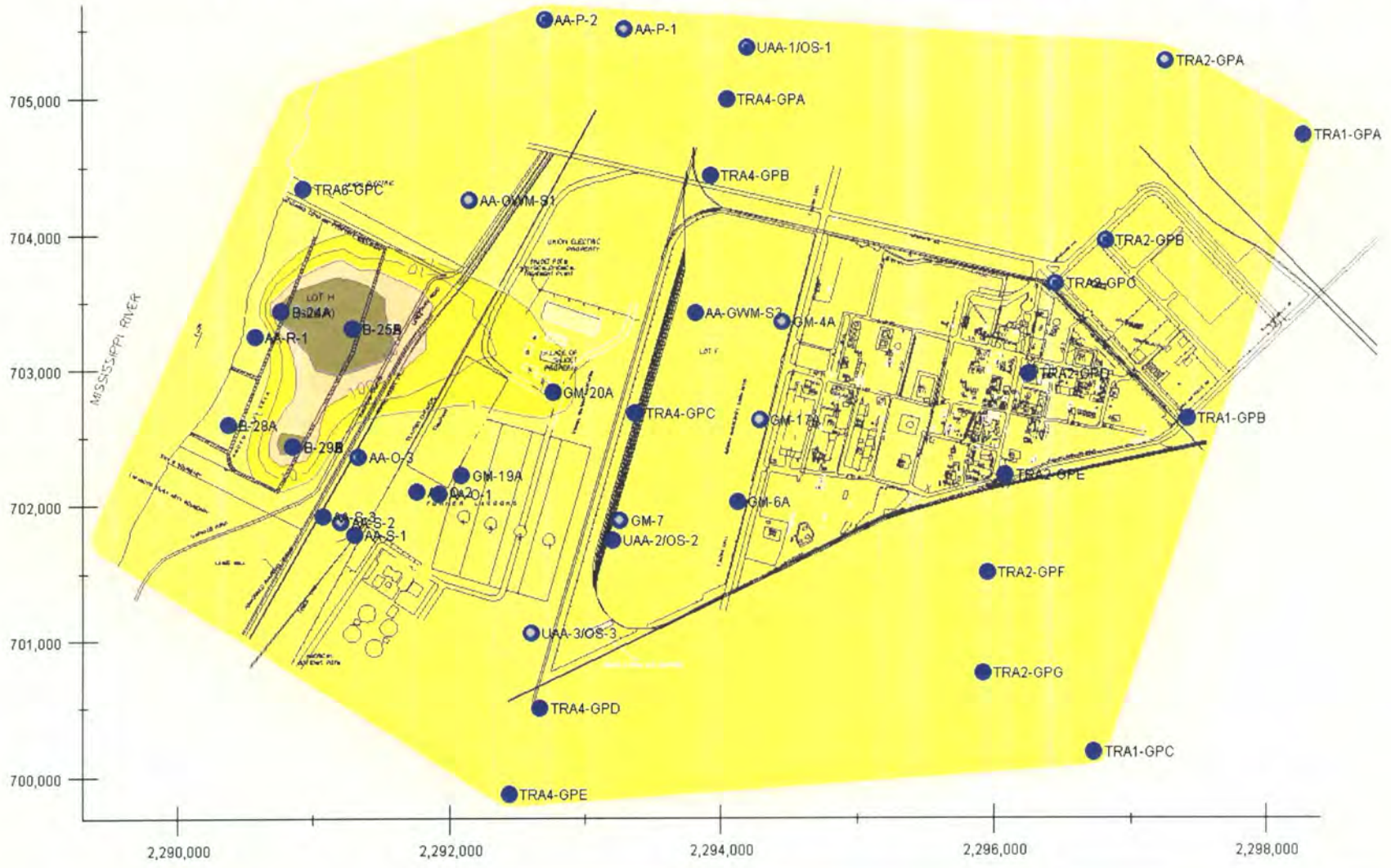
Date: 07/14/2005	
Figure Number: B-15	
Project Number: 21561601.00000	

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of 2,4-Dichlorophenol in Groundwater – SHU (Elevation 395 to 380)



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005	
Figure Number: B-16	
Project Number: 21561601.00000	

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of 2,4-Dichlorophenol in Groundwater – MHU (Elevation 380 to 350)

<1 ug/l

1 to 10 ug/l

10 to 100 ug/l

100 to 1000 ug/l

1000 to 10000 ug/l

>10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: B-17

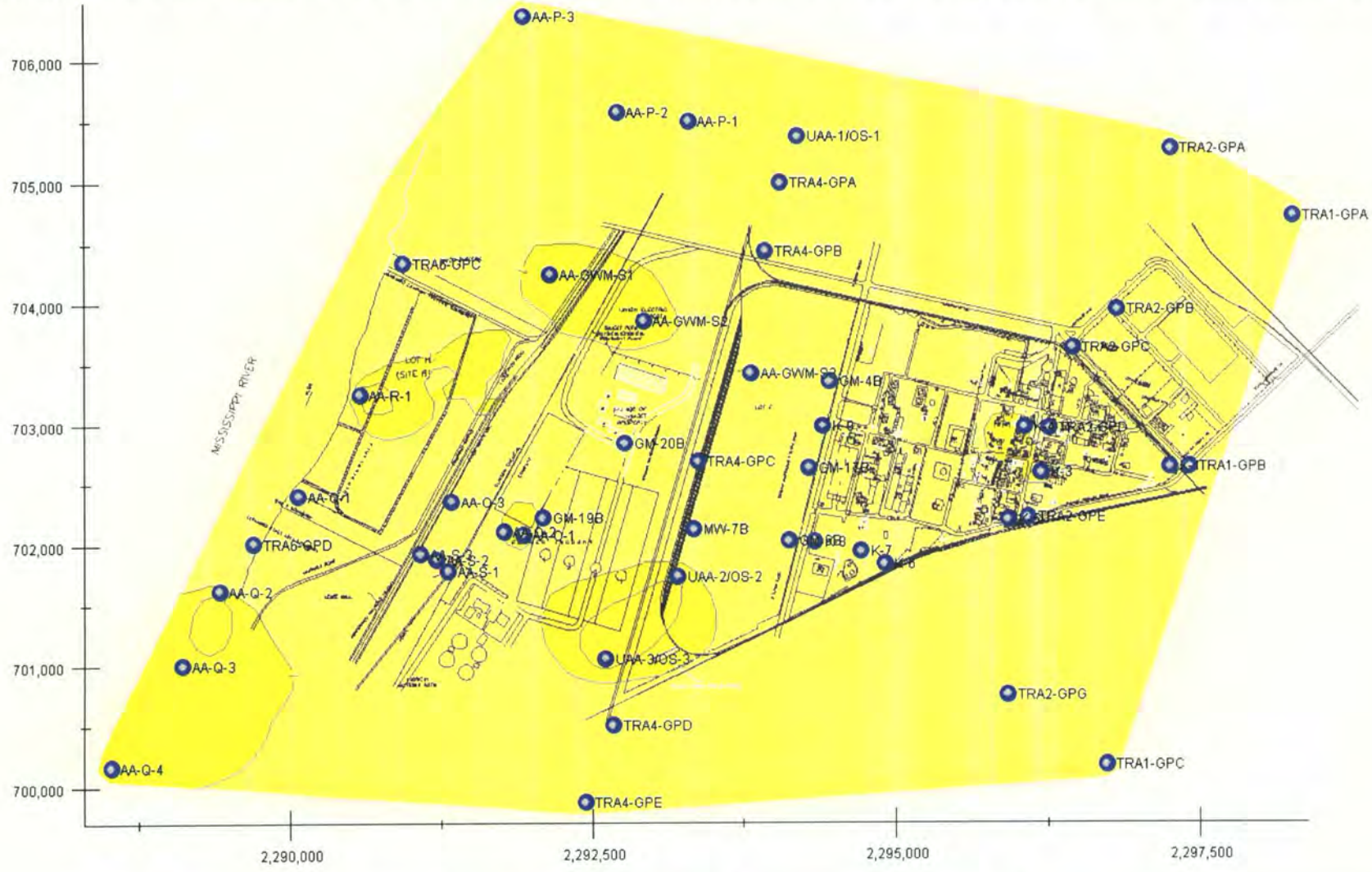
Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of 2,4-Dichlorophenol in Groundwater – DHU (Elevation 350 to Bedrock)



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005	
Figure Number: B-18	
Project Number: 21561601.00000	

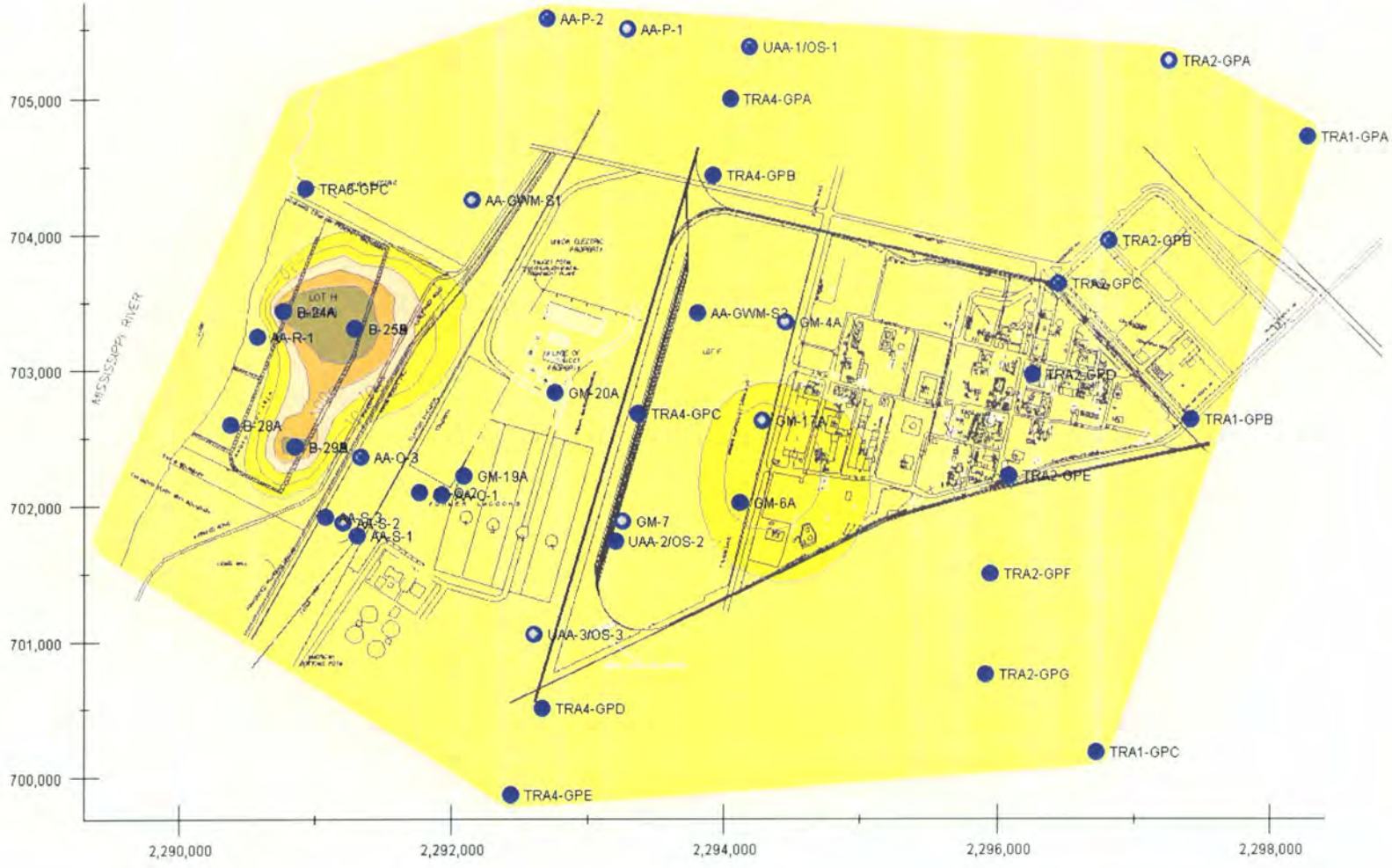
Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

Maximum Concentration of Phenol in Groundwater – SHU (Elevation 395 to 380)

URS

N



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005	
Figure Number: B-19	
Project Number: 21561601.00000	

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

Maximum Concentration of Phenol in Groundwater – MHU (Elevation 380 to 350)

URS

<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005	
Figure Number: B-20	
Project Number: 21561601.00000	

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

Maximum Concentration of Phenol in Groundwater – DHU (Elevation 350 to Bedrock)

URS

<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

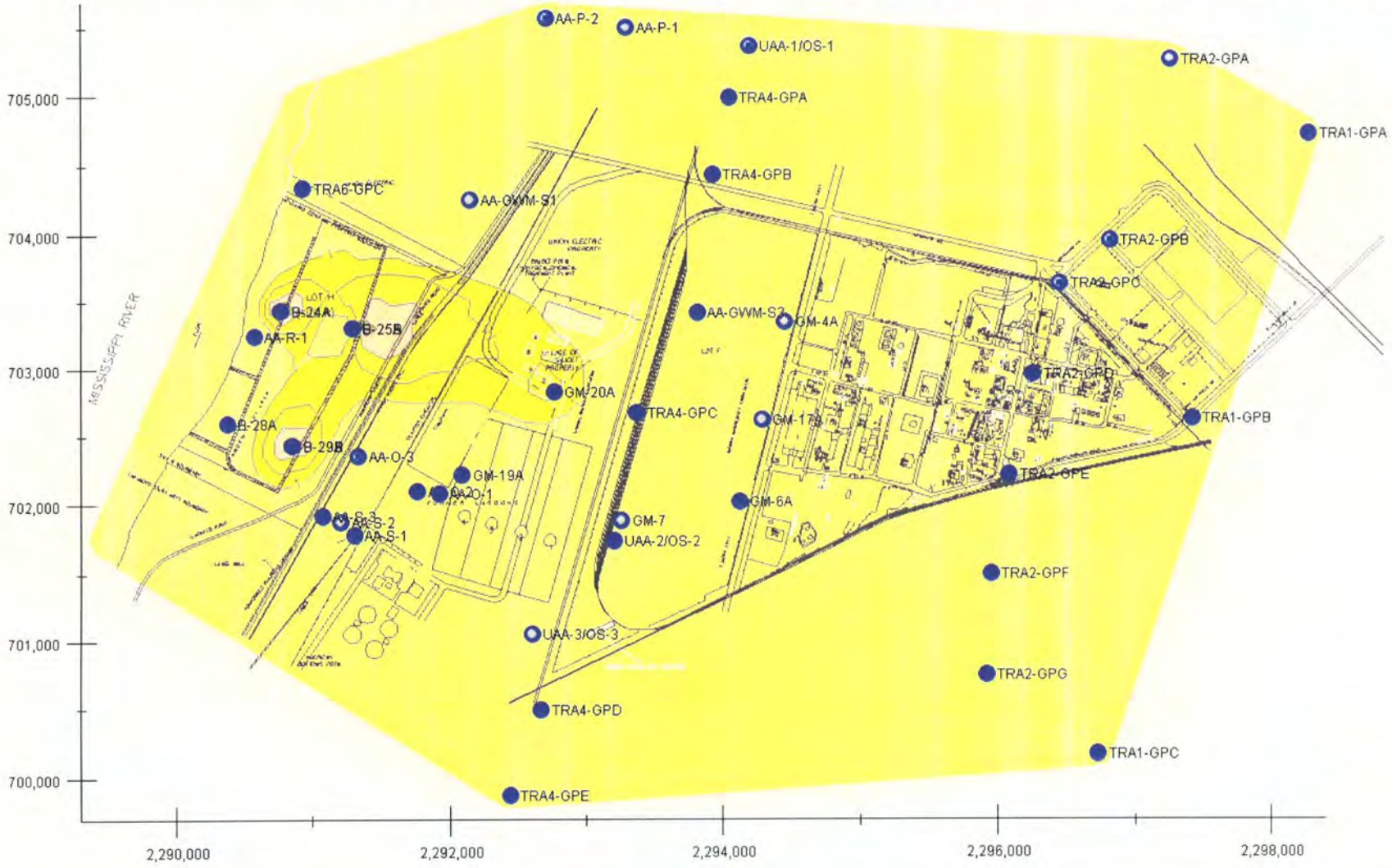
Date: 07/14/2005	
Figure Number:	B-21
Project Number:	21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of 2,4,6-Trichlorophenol in Groundwater – SHU (Elevation 395 to 380)



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005	
Figure Number:	B-22
Project Number:	21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of 2,4,6-Trichlorophenol in Groundwater – MHU (Elevation 380 to 350)

<1 ug/l
1 to 10 ug/l
10 to 100 ug/l
100 to 1000 ug/l
1000 to 10000 ug/l
>10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: B-23

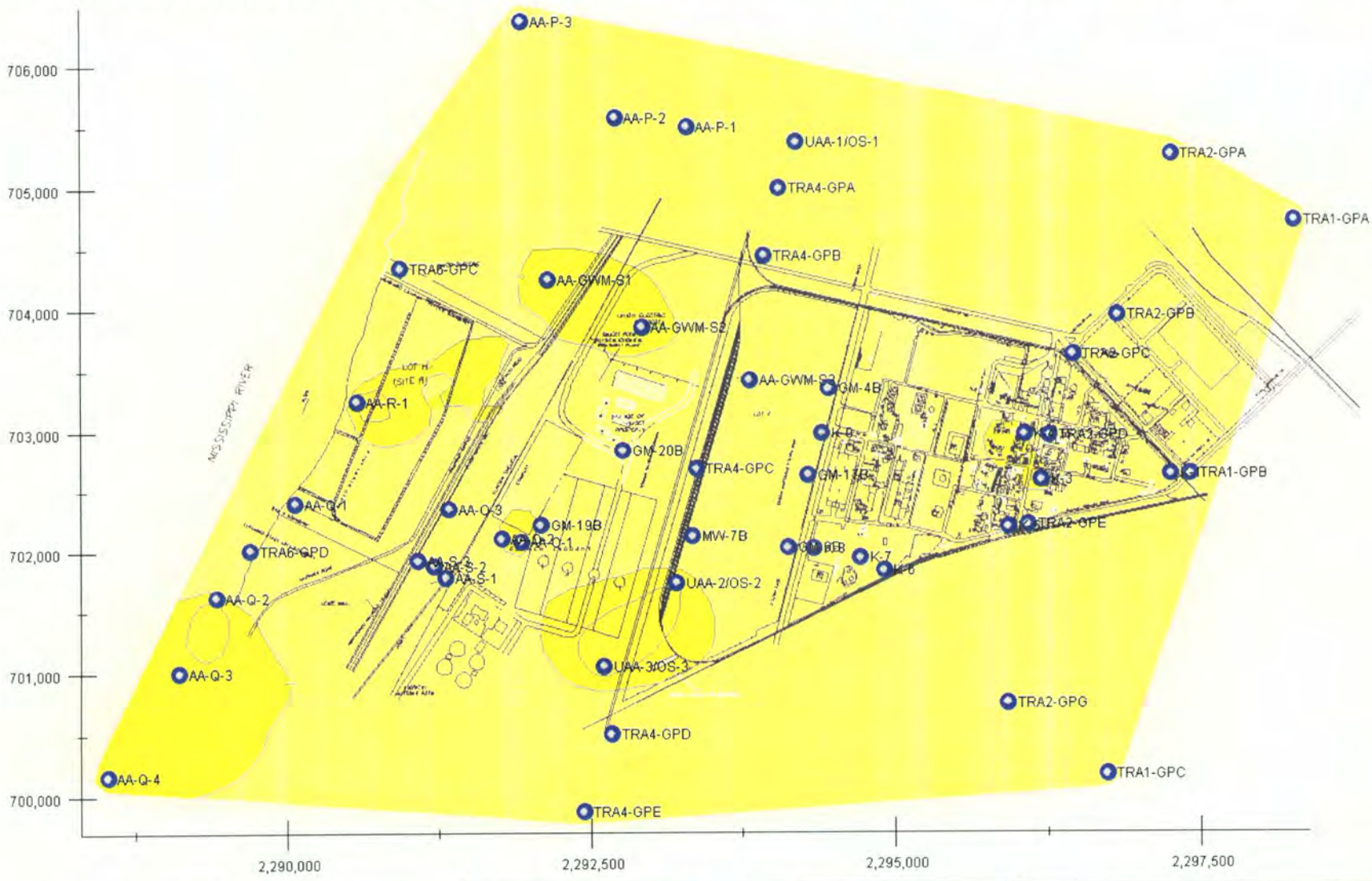
Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of 2,4,6-Trichlorophenol in Groundwater – DHU (Elevation 350 to Bedrock)



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005

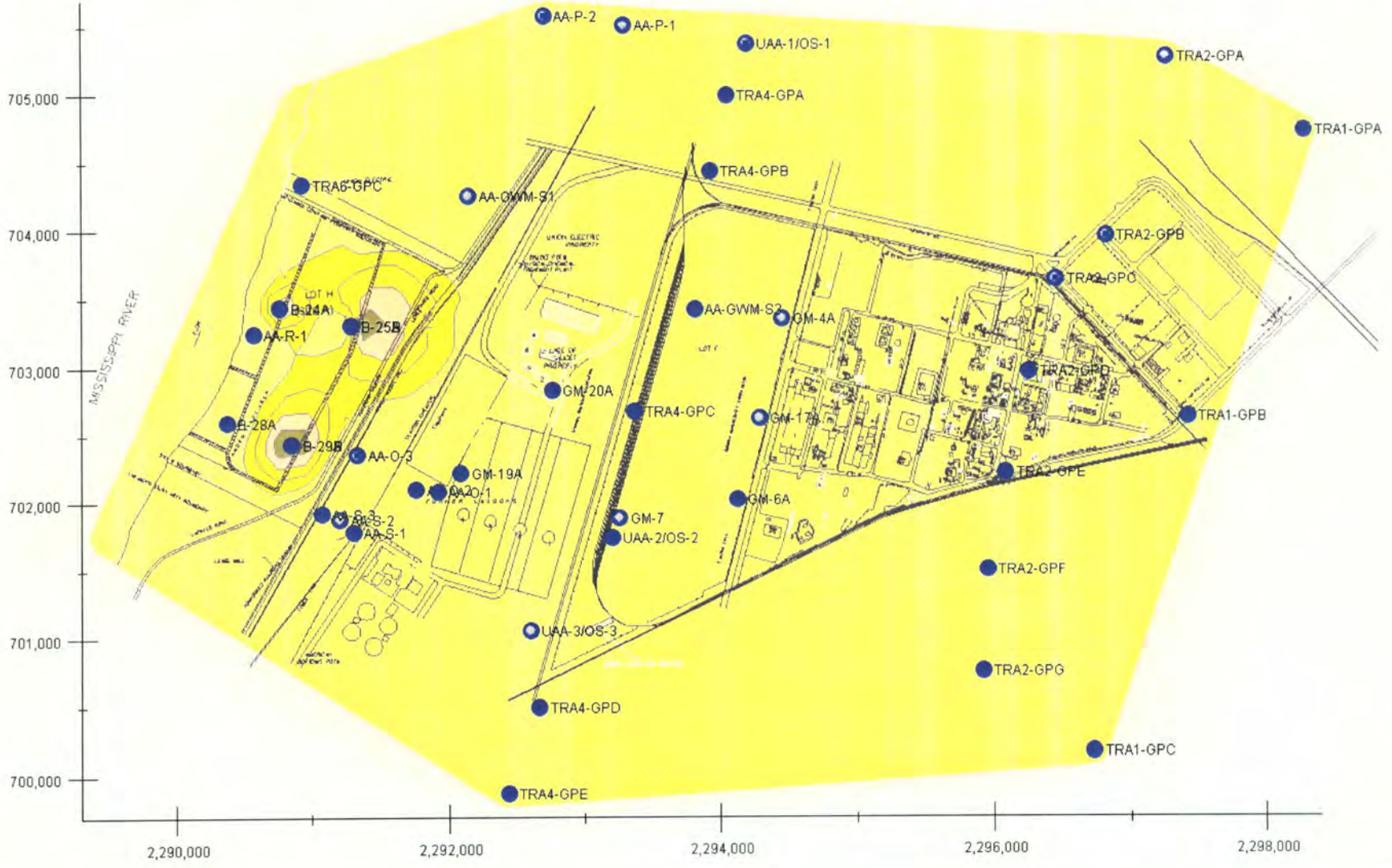
Figure Number: B-24

Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View Maximum Concentration of Methylphenols in Groundwater – SHU (Elevation 395 to 380)

URS



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005	
Figure Number:	B-25
Project Number:	21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of Methylphenols in Groundwater – MHU (Elevation 380 to 350)

- <1 ug/l
- 1 to 10 ug/l
- 10 to 100 ug/l
- 100 to 1000 ug/l
- 1000 to 10000 ug/l
- >10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: B-26

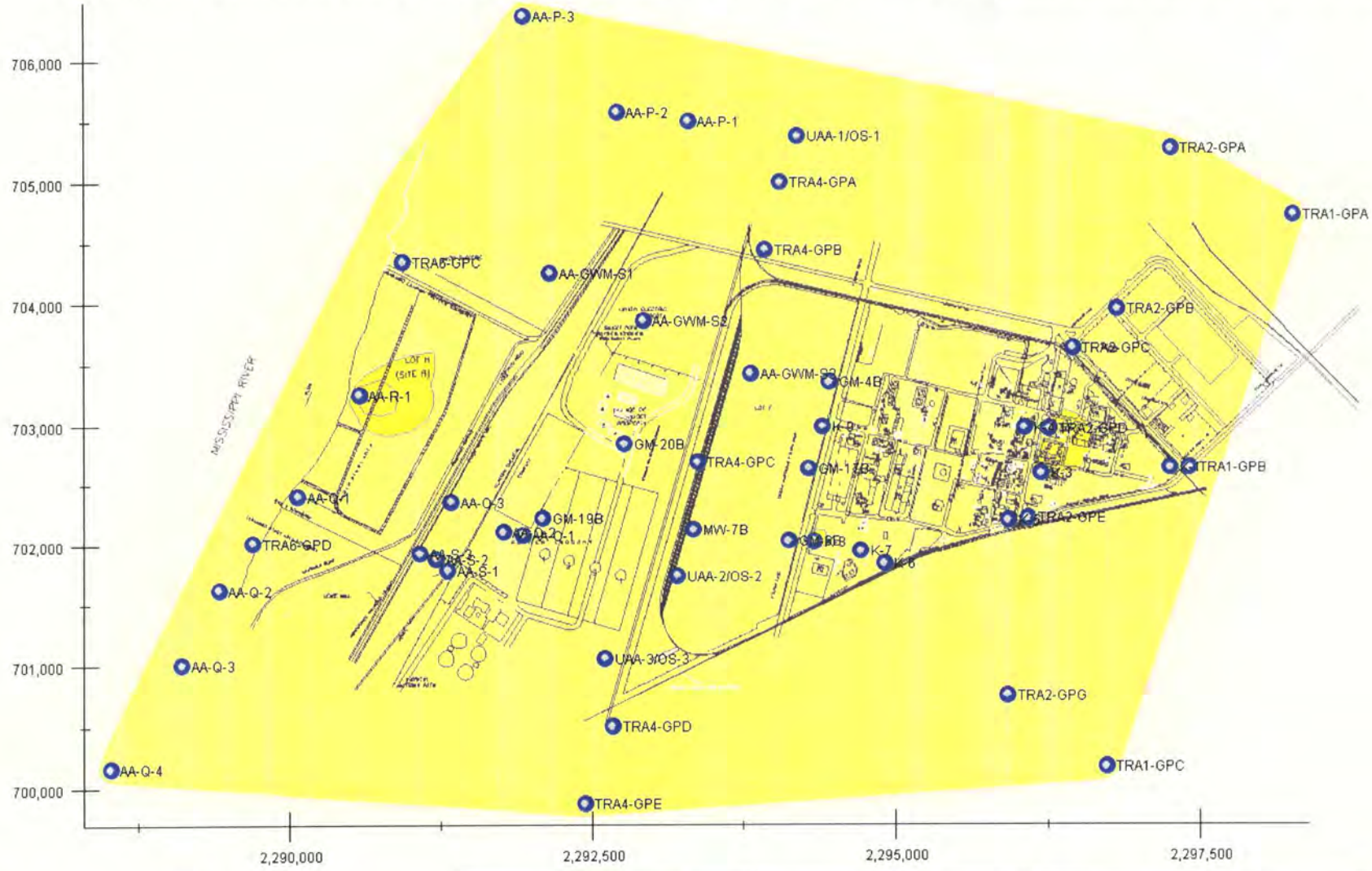
Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of Methylphenols in Groundwater – DHU (Elevation 350 to Bedrock)



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

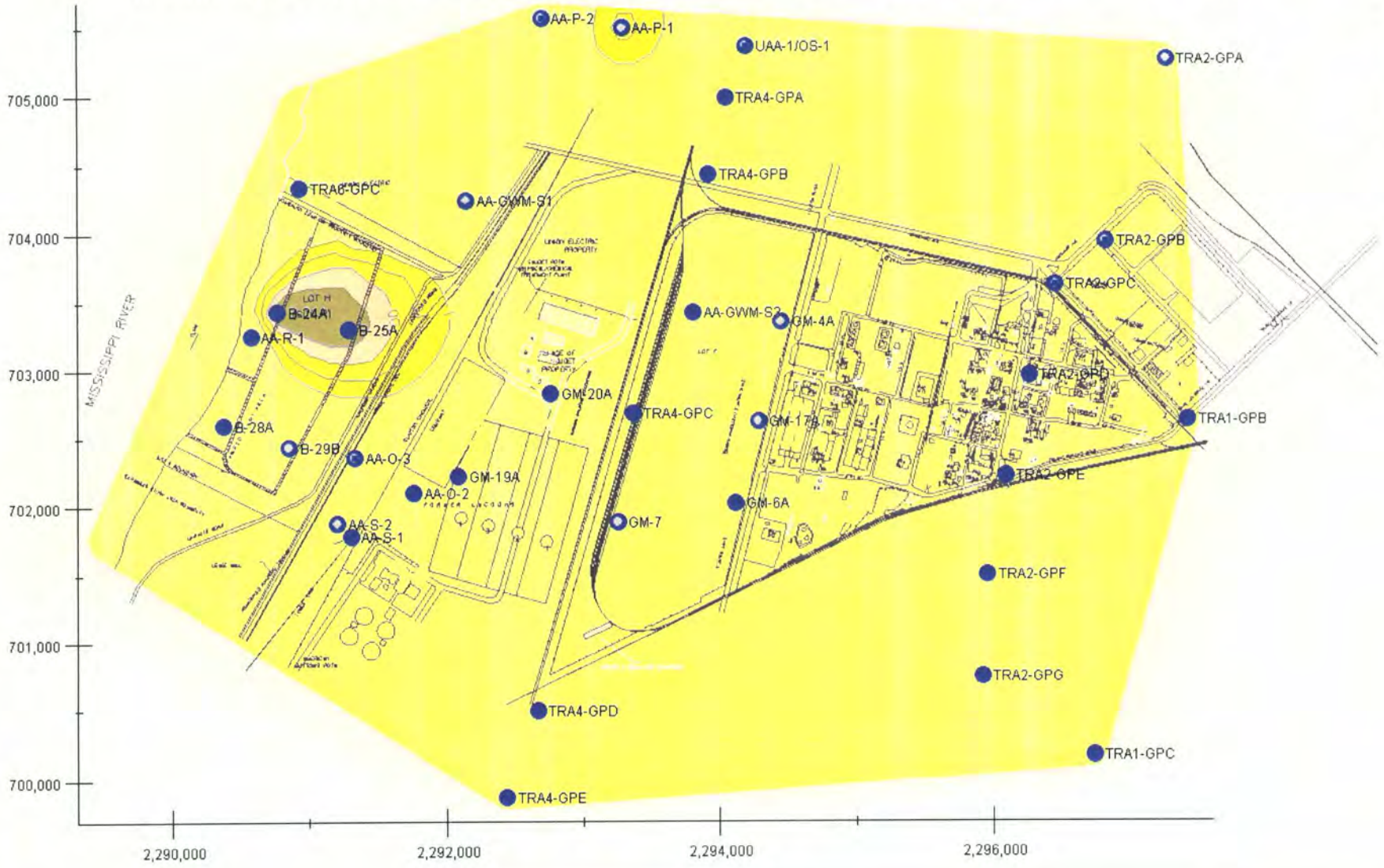
Date: 07/14/2005	
Figure Number:	B-27
Project Number:	21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

Maximum Concentration of Acetone in Groundwater – SHU (Elevation 395 to 380)

URS



<1 ug/l
1 to 10 ug/l
10 to 100 ug/l
100 to 1000 ug/l
1000 to 10000 ug/l
>10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: B-28

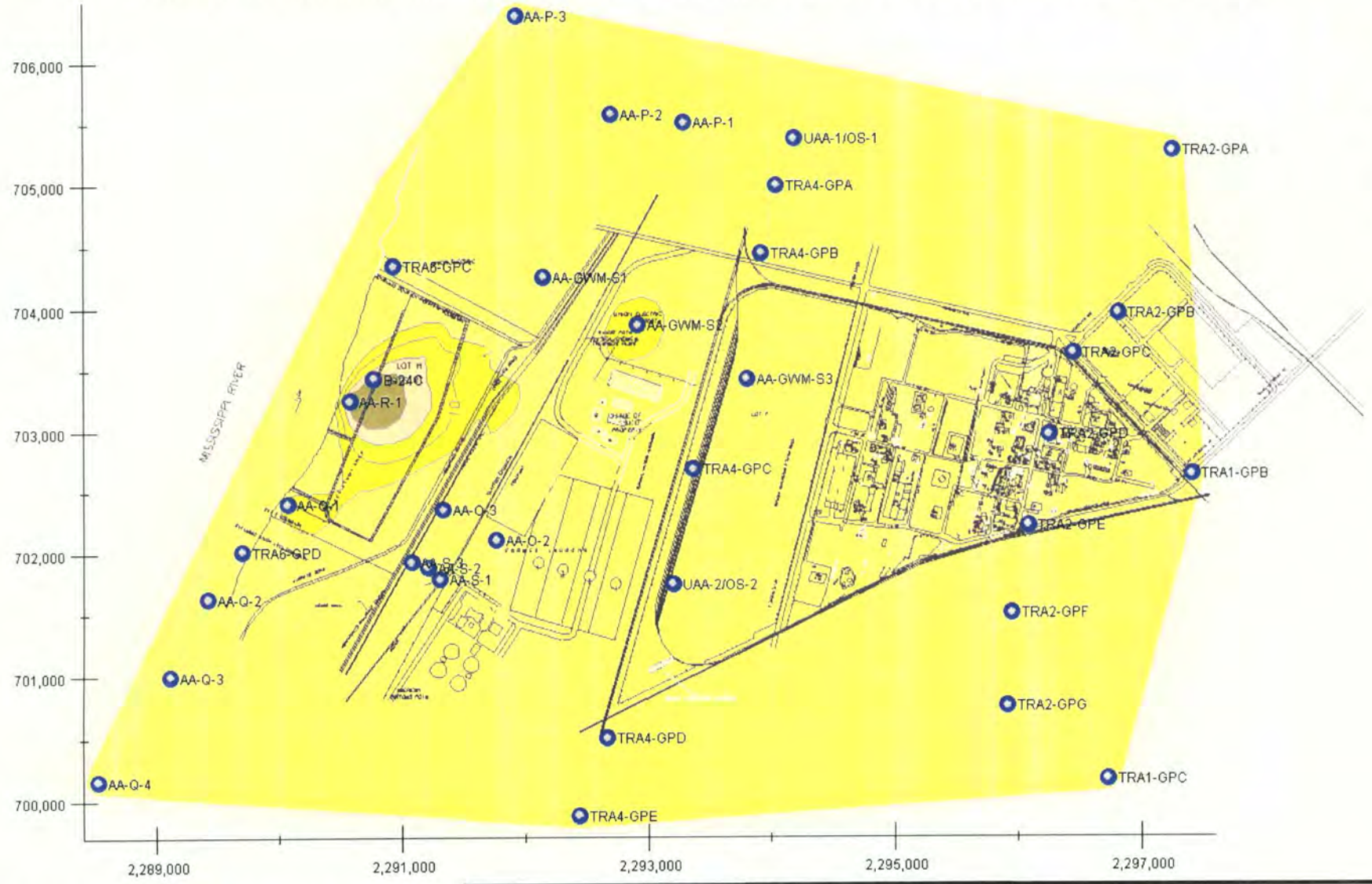
Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

Maximum Concentration of Acetone in Groundwater – MHU (Elevation 380 to 350)

URS



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005

Figure Number: B-29

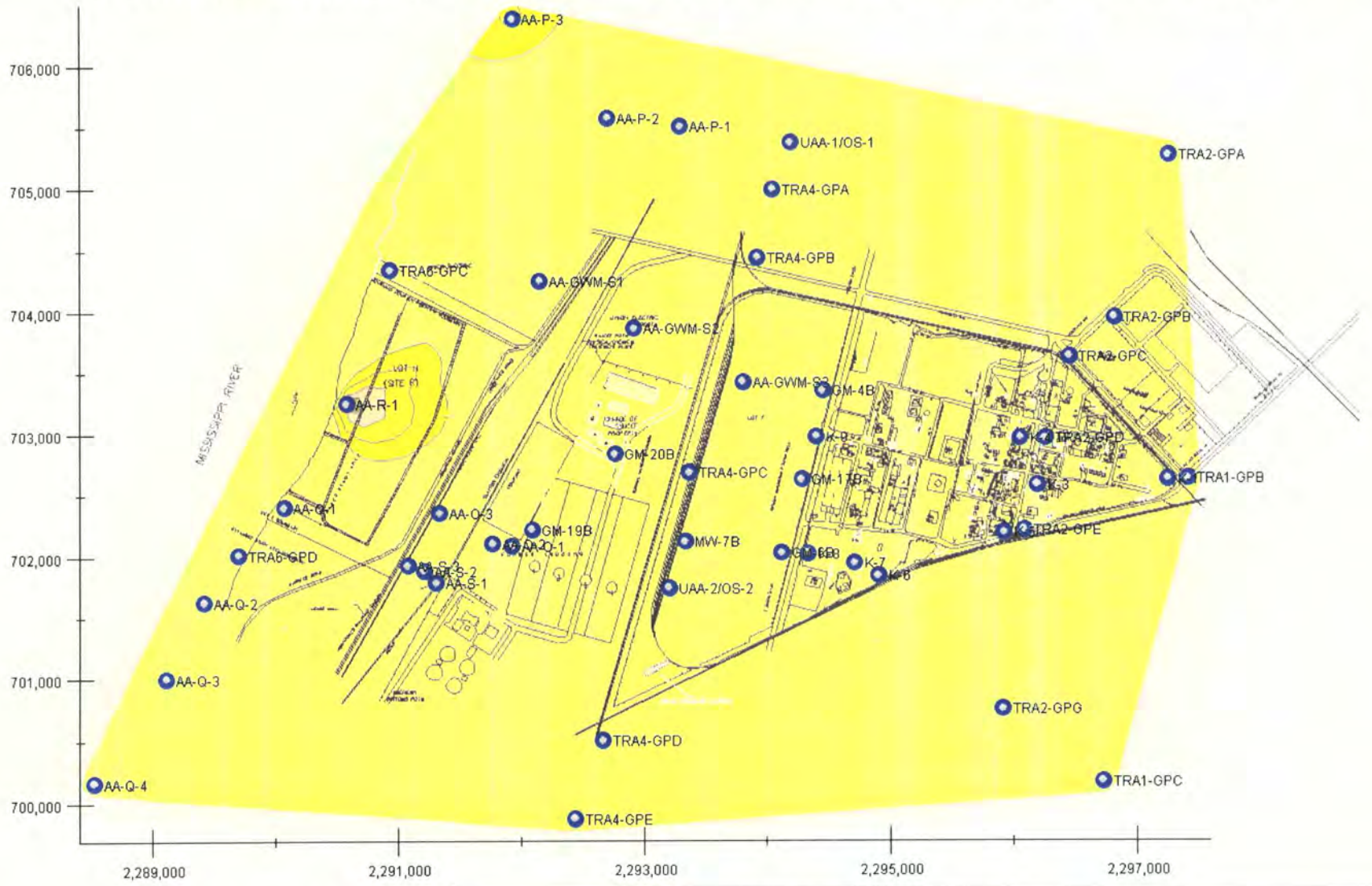
Project Number: 21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

URS

Maximum Concentration of Acetone in Groundwater – DHU (Elevation 350 to Bedrock)



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

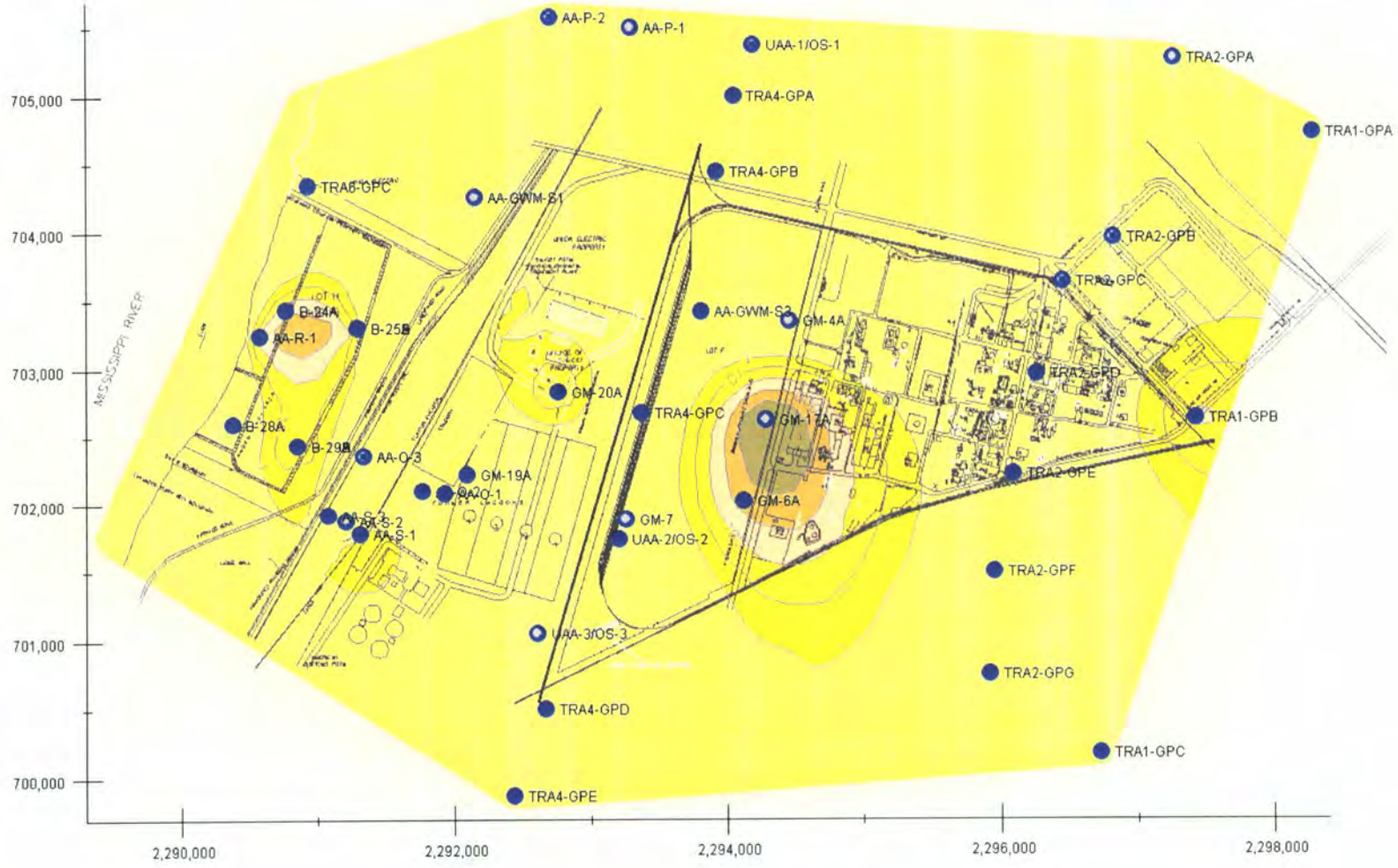
Date: 07/14/2005	
Figure Number:	B-30
Project Number:	21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

Maximum Concentration of BTEX in Groundwater – SHU (Elevation 395 to 380)

URS



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005	
Figure Number: B-31	
Project Number: 21561601.00000	

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

Maximum Concentration of BTEX in Groundwater – MHU (Elevation 380 to 350)

URS

<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

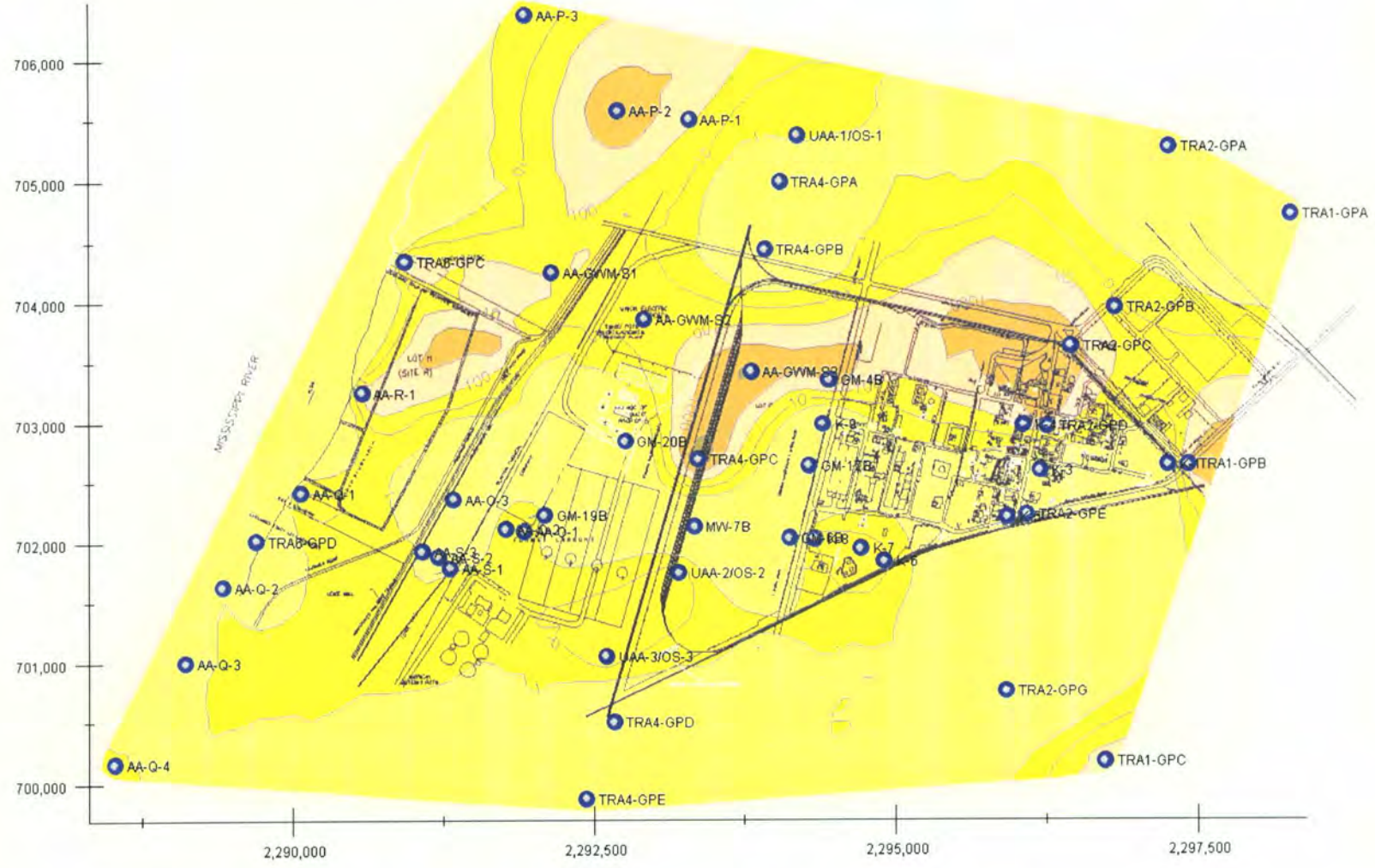
Date: 07/14/2005	
Figure Number:	B-32
Project Number:	21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Plan View

Maximum Concentration of BTEX in Groundwater – DHU (Elevation 350 to Bedrock)

URS



<1 ug/l	100 to 1000 ug/l
1 to 10 ug/l	1000 to 10000 ug/l
10 to 100 ug/l	>10000 ug/l

SOLUTIA

Date: 07/14/2005	
Figure Number:	B-33
Project Number:	21561601.00000

Plume Stability Monitoring Plan
W.G. Krummrich Facility
Sauget, Illinois

Field Standard Operating Procedures

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to define the procedures and necessary equipment for installation of groundwater monitoring wells and piezometers. A piezometer is simply a small diameter monitoring well. Therefore, the equipment and procedures for constructing a piezometer are the same as those used to install any monitoring well. The step-by-step procedures described herein are sufficiently detailed to allow field personnel to properly install any size monitoring well (or piezometer).

2.0 EQUIPMENT NEEDED

This section details the required equipment for the drilling and installation of groundwater monitoring wells.

The following is an equipment list typical for well drilling and installation:

- Drill rig capable of installing wells to the desired depth in the expected formation materials and conditions
- Well casing and well screen
- Bentonite pellets or chips
- Filter pack sand
- Portland Type I or II Cement and powdered bentonite for grouting
- Protective well casing with locking cap
- High-pressure steamer/cleaner
- Long-handled bristle brushes
- Wash/rinse tubs
- Appropriate decontamination supplies
- Location map
- Plastic bags (Ziploc)
- Self-adhesive labels
- Weighted tape measure
- Water level probe
- Deionized or distilled water

- Appropriate health and safety equipment as specified in the Health and Safety Plan (HSP)
- Log book
- Boring log sheets
- Well construction form
- Plastic sheeting
- Drums for containment of cuttings and Decontamination and/or development water (if necessary).

3.0 PROCEDURES

Decontamination

Before drilling or well installation begins, all drilling and well installation material will be decontaminated according to the protocols listed in the decontamination SOP. Drilling equipment will be decontaminated between well locations.

Instrument Calibration

Before going into the field, the sampler shall verify that field instruments are operating properly. Calibration times and readings will be recorded in a notebook to be kept by the field sampler. Specific instructions for calibrating the instruments are given in the respective SOPs.

Drilling and Well Installation Procedures

Drilling Technique

If soil sampling is required, all soil samples will be taken following the protocol outlined in the soil sampling SOP. The hole will be logged following the methods specified in the logging SOP.

Boreholes will be advanced using drilling methods specified in the Work Plan and a drill rig capable of completing the monitor well(s) to the depth(s) specified in the Scope of Work. Before drilling, well locations will be numbered and staked. The necessary permits and utility clearances will be obtained prior to commencement of drilling activities. Appropriate health and safety measures will be followed during drilling and well installation activities as specified in the Health and Safety Plan.

During the drilling operation, the cuttings from the boring will be containerized or placed directly onto the ground as specified in the Work Plan. Disposal of cuttings will be in accordance with the Work Plan.

Monitor Well Drilling Operations

The procedures for drilling are as follows:

- Set up drilling rig at staked and cleared borehole location.
- Record location, date, time and other pertinent information in the field book.
- Drill hole of appropriate size using hollow-stem augers.
- Collect samples at the predetermined intervals, if appropriate, for sample description and/or chemical analysis as specified in the plan.
- Complete the borehole to the depth specified in the Work Plan.

Well Design Specifications

The following general specifications will be:

Boring Diameter: The boring will be of sufficient diameter to permit at least two inches of annular space between the boring wall and all sides of the centered riser and screen. The boring diameter will be of sufficient size to allow for the accurate placement of the screen, riser, filter pack, seal, and grout.

Well Casing: Well riser will consist of new threaded, flush joint, PVC or stainless steel. Well diameter and thickness will be specified in the Work Plan. Risers will extend approximately two feet above the ground surface, except in the case of flush-mount surface casings (see Work Plan for appropriate construction). The tops of all well casings will be fitted with expandable locking caps or PVC slip caps.

Well Screens: Screen length for each well will be specified in the Work Plan. Well screens will consist of new threaded pipe with factory-machine slots/ wrapped screen with an inside diameter equal to or greater than that of the well casing. The slot size will be indicated in the Work Plan and designed to be compatible with aquifer and sand pack material. The schedule thickness of PVC screen will be the same as that of the well casing. All screen bottoms will be fitted with a cap or plug of the same composition as the screen and should be within 0.5 foot from the open part of the screen. Traps may be used.

Well Installation Procedure

The following procedures will be initiated within 12 consecutive hours of boring completion for uncased holes or partially cased holes and within 48 consecutive hours for fully cased holes. Once installation has begun, no breaks in the installation procedure will be made if no unusual conditions are encountered until the well has been grouted and the drill casing has been removed.

The procedure for monitoring well installation is as follows:

1. Decontaminate all well materials according to the decontamination SOP. Following decontamination, all personnel that handle the casing will don a clean pair of rubber or surgical gloves.
2. Measure each section of casing, and screen, to nearest 0.10 foot.
3. Assemble screen and casing as it is lowered into the borehole.
4. Lower screen and casing to about 6 inches above the bottom of the boring.
5. Record the level of top of casing and calculate the screened interval. Adjust screen interval by raising assembly to desired interval if necessary and add sand to raise the bottom of the boring.
6. Calculate and record the volume of the filter pack, bentonite seal, and grout required for existing boring conditions.
7. Begin adding filter pack sand around the annulus of the casing a few feet at a time. Repeated depth soundings shall be taken to monitor the level of the sand.
8. Allow sufficient time for the filter sand to settle through the water column outside the casing before measuring the sand level.
9. Extend the filter pack sand to at least 2 feet above the top of the well screen.
10. Following sand filter pack placement in the shallow wells, install a minimum 3 foot-thick seal of bentonite pellets or chips by slowly adding the pellets to avoid bridging. The thickness of the completed bentonite seal shall be measured before the pellets are allowed to swell. The completed bentonite seal shall be allowed to hydrate before proceeding with the grouting operations.
11. Grout the remaining annulus from the top of the bentonite seal to about 3 feet below the surface as measured after the augers are removed. The grout will be tremied into the borehole until the annulus is completely filled. The base of the tremie pipe should be placed approximately 5 feet above the bentonite seal.
12. After the grout sets for 24 hours it will be checked for settlement. If necessary, additional grout will be added to top off the annulus.
13. The concrete pad and bollards, if required, will be installed according to specifications in this SOP. The protective casing and posts will be painted a high visibility color.

14. Optional - URS personnel will affix a permanent, non-corrosive tag to the outer steel protective casing of each well which clearly identifies the well number.

Well Installation Specifications:

Filter Pack: The annular space around the well screen will be backfilled with a clean, washed, silica sand sized to perform as a filter between the formation material and the well screen. The filter pack will extend a minimum two feet above the screen and may be tremied into place. The final depth to the top of the filter pack will be measured directly using a weighted tape measure or rod and not by using volumetric calculation methods. The grain size of the filter pack will be shown on the well construction log.

Bentonite Seal and Grout: A three-foot thick (minimum) bentonite pellet/slurry seal will be placed in the annulus above the filter pack. The thickness of the seal may vary slightly based on site conditions. The thickness of the seal will be measured immediately after placement, without allowance for swelling. Bentonite slurry seals should have a thick batter-like consistency. Slurry seals will have a maximum placement thickness of 8 feet. Cement grout will then be placed from the top of the bentonite seal to the ground surface. The cement grout will consist of a mixture of Portland cement (ASTM C150) and clean water in the proportion of not more than seven gallons of clean water per bag of cement (one cubic foot or 94 pounds). Additionally, three percent by weight of bentonite powder will be added if permitted by state regulations. The grout will be prepared in an above-ground rigid container by first thoroughly mixing the cement with water and then mixing in the bentonite powder. The grout will be placed by pumping through a tremie pipe. The lower end of the tremie pipe will be kept within five feet of the top of the bentonite seal. Grout will be pumped through the tremie pipe until undiluted grout flows from the annular space at the ground surface. The tremie pipe will then be removed and more grout added to compensate for settling. After 24 hours, the drilling contractor will check the site for grout settlement and add more grout to fill any depression. This will be repeated until firm grout remains at the surface.

Protection of Well: URS personnel will at all times during the progress of the work, take precautions to prevent tampering with the wells or entrance of foreign material into them. Upon completion of a well, a suitable cap will be installed to prevent foreign material from entering the well. The wells will be enclosed in a steel protective casing. Steel casings will be, at a minimum, 4 inches in diameter and will be provided with locking caps and locks. All locks will be keyed alike. If the well is to be a stickup, as specified in the Work Plan, a 1/4-inch drainage hole will be drilled in the protective steel casing centered approximately 1/8-inch above the internal mortar collar for drainage. The well designation will be painted on the

protective casing, using a brush. Painting will be done prior to well development. If specified in the Work Plan, a minimum 2-foot by 2-foot, 6 to 8-inch-thick concrete pad, sloped away from the well, will be constructed around the protective casing at the final ground level elevation. Three or four 2-inch-diameter or larger steel posts will be equally spaced around the well and embedded in separate concrete filled holes just outside of the concrete pad. The protective steel posts will extend approximately 1 foot above the well riser. Any well that is to be temporarily removed from service or left incomplete due to delay in construction, will be capped with a water tight cap and equipped with a "vandal-proof" cover satisfying applicable state or local regulations or recommendations.

4.0 DOCUMENTATION

Observations and data acquired in the field during drilling and installation of wells will be recorded to provide a permanent record. A boring log will be completed for each boring according to the procedures outlined in the logging SOP.

Additional documentation for well construction will be written in the field book according to the field reporting and documentation SOP and will include the following:

- Date
- Time
- Personnel
- Weather
- Subcontractors
- Health and Safety monitoring equipment and readings
- Grout, sand, and bentonite volume calculations prior to well installation
- The quantity and composition of the grout, seals, and filter pack actually used during construction
- Screen slot size (in inches), slot configuration, outside diameter, nominal inside diameter, schedule/thickness, composition, and manufacturer
- Coupling/joint design and composition
- Protective casing composition and nominal inside diameter
- Start and completion dates

- Discussion of all procedures and any problems encountered during drilling and well construction.

In addition, the well installation details will be shown in a diagram which will be drawn in the field book. Each well diagram will consist of the following (denoted in order of decreasing depth from ground surface):

- Reference elevation for all depth measurements
- Project and site names
- Well number
- Date(s) of installation
- The depth at which the hole diameter changes (if appropriate)
- The depth of the static water level and date of measurement(s)
- Total depth of completed well
- Depth of any grouting or sealing
- Nominal hole diameter(s)
- Amount of cement used for grouting or sealing
- Depth and type of well casing
- Description (to include length, internal, diameter, slot size, and material of well screen(s)
- Any sealing off of water-bearing strata
- Static water level upon completion of the well and after development
- Drilling date(s)
- Other construction details of monitoring well including grain size of well filter pack material and location of all seals and casing joints.

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) describes the methods to be used in measuring organic vapors emitted from soils collected with a mechanical device or hand augering device. Results will be used as a field screening for volatile organic vapors.

2.0 PURPOSE

The purpose of this procedure is to maintain uniformity between field personnel performing the measurements and to provide representativeness of readings obtained.

3.0 EQUIPMENT NEEDED

Personal protective equipment, PID, wide-mouth sample jars and aluminum foil or polyethylene bags (Ziploc type), rubber bands, field data forms.

4.0 PROCEDURES

1. Samples are collected and placed in wide-mouth sample jars or polyethylene bags (zip-loc type) so that the jars or bags are approximately half full. The jars or bags are labeled to document sample location and depth, time, date, and field personnel collecting the sample.
2. The glass jar is capped with aluminum foil, a rubber band, and the lid, if it will fit or the bag is zipped shut.
3. The air-tight sample container is then allowed to warm for at least 10-15 minutes to allow the liberation of soil gases into the headspace.
4. Calibrate and prepare PID for use.
5. Puncture the aluminum foil or polyethylene bag with the calibrated monitor probe and allow headspace gases to be drawn through the PID unit.
6. Record the highest response obtained on an appropriate sampling log.
7. Remove the punctured foil and seal jar with the proper lid.
8. Allow instrument to return to zero and repeat procedure for next sample.

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) outlines the procedures for soil sample logging. The purpose of soil sample logging is to record all field notes in a detailed and organized fashion.

2.0 PROCEDURES

At the outset of sample logging, the on-site geologist will record field notes with waterproof ink in a bound field notebook. At a minimum, the daily field notes will include:

- Project name and number
- Date and time
- Weather conditions
- Sampler's name
- Project objective(s).

Throughout the sampling round, the following items will be recorded as appropriate:

- Sample location(s)
- Sample identifications
- Limiting field conditions
- Problems encountered.

Unconsolidated soil samples will be described as follows:

- Descriptive information:
  - Color name (Munsell Color Chart) of the logged interval or sample
  - Color notation including chroma, hue, value, and qualifiers

1. Mottling with abbreviations, descriptors, and criteria for descriptions of mottles as identified below

Descriptors for Mottling

Abundance	Size	Contract
f: few (<2%)	fine (<5 mm)	faint
c: common (2%-20%)	medium (5-15 mm)	distinct
m: many (>20%)	coarse (>15 mm)	prominent

2. Degree of saturation (dry, damp, moist, wet, saturated, or combinations); note depth to groundwater table, if observed

3. Degree of density. Count the blows of each 12-inch increment of the sampler (ASTM-1586-84). Use the values and the density table presented below to determine the degree of density.

Degree of Density

Cohesive Clays		Non-cohesive Granular Soils	
<2	very soft	0-4	very loose
2-4	soft	>4-10	loose
>4-8	medium stiff	>10-30	medium dense
>8-15	stiff	>30-50	dense
>15-30	very stiff	>50	very dense
>30	hard		

4. Soil description according to ASTM's Unified Soil Classification System (USC) and by soil structure:

   - ASTM Unified Soil Classification: The Grade Limits and Grade Standards table presented below provides the grade limits and grade names used by engineers according to ASTM standards D422-63 and D643-78.

Grade Limits and Grade Standards

Grade Limits		Grade Names
mm	inch	US standard sieve series
		boulders
305	12.0	
		cobbles
76.2	3.0	3.0 inch
		gravel
4.75	0.19	No. 4
2.00	0.08	No. 10
		medium sand
0.425		No. 40
0.074		No. 200
		silt
0.005		
		clay size

Source: AGI data sheet 29.2

   - Course-grained soils include clean gravels and sands and silty or clayey gravels and sands with more than 50% retained on the No. 200 sieve. A table of USC symbols and names for coarse-grained soils is presented below.

USCS Symbols and Names for Coarse-grained Soils

USCS Symbol	Typical Names
GW	Well graded gravels, gravel-sand mixtures, little or no fines
GP	Poorly graded gravels, gravel-sand mixtures, little or no fines
GM	Silty gravels, gravel-sand-silt mixtures
GC	Clayey gravels, gravel-sand-clay mixtures
SW	Well graded sands, gravelly sands, little or no fines
SP	Poorly graded sands, gravelly sands, little or no fines
SM	Silty sand, sand-silt mixtures
SC	Clayey sands, sand-clay mixtures

- Fine-grained soils include inorganic and organic silts and clays; gravelly, sandy, or silty clays; and clayey silts with more than 50% passing the No. 200 sieve. A table of USC symbols and names for fine-grained soils is presented here.

USCS Symbols and Names for Fine-grained Soils

USCS Symbol	Typical Names
ML	Inorganic silts and very fine sands, rock flour, silty, or clayey fine sands, or clayey silts with slight plasticity
CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays
OL	Organic silts and organic silty clays of low plasticity
MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts
CH	Inorganic clays or high plasticity (residual clays), fat clays
OH	Organic clays of medium to high plasticity, organic silts
Pt	Peat and other highly organic soils

- A table of soil descriptors is presented below.

Soil Descriptors

Calcareous:	containing appreciable quantities of calcium carbonate
Fissured:	containing shrinkage cracks, often filled with fine sand or silt, usually more less vertical
Interbedded:	containing alternating layers of different soil types
Intermixed:	containing appreciable, random, and disoriented quantities of varying color, texture, or constituency
Laminated:	containing thin layers of varying color, texture, or constituency
Layer:	thickness greater than 3 inches

Soil Descriptors

Mottled:	containing appreciable random speckles or pockets of varying color, texture, or constituency
Parting:	paper thin
Poorly graded (well sorted):	primarily one grain size, or having a range of sizes with some intermediate size missing
Slickensided:	having inclined planes of weakness that are slick and glossy in appearance and often result in lower unconfined compression cohesion
Split graded:	containing two predominant grain sizes with intermediate sizes missing
Varved:	sanded or layered with silt or very fine sand (cyclic sedimentary couplet)
Well graded (poorly sorted):	containing wide range of grain sizes and substantial amounts of all intermediate particle sizes
Modifiers:	Predominant type - 50% to 100% Modifying type - 12% to 50% With - 5% to 12% Trace - 1% to 5%

5. Degree of plasticity. The following table presents the terms used to denote the various degrees of plasticity of soil that passes the No. 200 sieve.

Degrees of Plasticity

Descriptive Term	Degree of Plasticity	Plasticity Index Range
SILT	none	non-plastic
Clayey SILT	slight	1-5
SILT & CLAY	low	5-10
CLAY & SILT	medium	10-20
Silty CLAY	high	20-40
CLAY	very high	over 40

6. Drilling information:

   - Drill rig manufacturer, model, and driller (if applicable)
   - Geologist or geotechnical engineer
   - Project name, sample point identification, and location
   - Date samples obtained (and times if required)
   - Type of sampler (e.g., split spoon, Shelby, California), measurements or method of advancing boring or equipment, method of driving sampler, and weight of hammer
   - Drill fluids (if applicable)

- Ground surface or grade elevation (if known)
- Depth penetrated and blow counts/6-inch interval of penetration for ASTM 1586-84 and sample number (if applicable)
- Closed hole intervals and advancement (if applicable)
- Recovery
- Strata changes and changes within samples
- Sampling tool behavior
- Drill string behavior
- Use(s) of borehole
- Disposition(s) of residual soil or cuttings
- Signature or sampling of log (as required)

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) describes the methods to be used for the calibration and use of the Photoionization Detector (PID) for field headspace analysis and health and safety monitoring.

2.0 PURPOSE

The purpose of this procedure is to develop and maintain good quality control in field operations and to create uniformity between field personnel involved with PID use.

3.0 EQUIPMENT NEEDED

PID (probes with 11.7 eV lamp or equivalent) or suitable for site conditions, log book, user's manual, calibration gas.

4.0 PROCEDURE

Calibration:

1. Prior to calibration, check the function switch on the control panel to make sure it is in the "OFF" position. The probe nozzle is stored inside the instrument cover. Remove cover plate by pulling up on the pins that fasten the cover plate.
2. Remove the nozzle from the cover. Assemble probe by screwing nozzle into casing.
3. Attach probe cable to instrument box inserting 12 pin interface connector of the probe cable into the connector on the instrument panel. Match the alignment keys and insert connector. Turn connector in clockwise direction until a distinct snap and lock is felt.
4. Turn the function switch to the Battery Check position. When the battery is charged, the needle should read within or above the green battery arc on the scale plate. If the needle is below the green arc or the red LED light comes on, the instrument should be recharged prior to making any measurements.
5. Turn the function switch to the "ON" position. In this position, the UV light source should be on. To verify, glance at the end of the probe for a purple glow. Do Not Look Directly at the Lamp Itself. If the lamp does not come on refer to the Instruction Manual.
6. To zero the instrument, turn the function switch to the standby position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counter clockwise rotation yields a downscale deflection. (Note: No zero gas is needed since this is an electronic zero adjustment.) If the span adjustment is changed during instrument calibration, the zero should be rechecked and adjusted. If necessary, wait 15 to 20 seconds to ensure that the zero reading is stable. Readjust as necessary.

Instrument Daily Calibration:

1. Insert one end of T-tube into probe. Insert second end of probe into calibration gas in the 20-200 ppm range. The third end of probe should have the rotometer (bubble meter) attached.
2. Set the function switch in the 0-200 ppm range. Crack the valve on the pressured calibration gas container until a slight flow is indicated on the rotameter. The instrument will draw in the volume required for detection with the rotameter indicating excess flow.
3. Adjust the span potentiometer so that the instrument is reading the exact value of the calibration gas. (Calibration gas value is labeled on the cylinder.)
4. Turn instrument switch to the standby position and check the electronic zero. Reset zero potentiometer as necessary following step 6 above.
5. Record all original and readjusted settings in log book.
6. Set the function switch to 0-20 ppm. Remove the mid-range (20-200 ppm) calibration gas cylinder and attach the low-range (0-20 ppm) calibration gas cylinder as described above.
7. Do not adjust the span potentiometer. The observed reading should be ± 3 ppm of the concentration specified for the low-range calibration gas. If this is not the case, recalibrate the mid-range scale repeating Steps 1 through 6 above. If the low-range reading consistently falls outside the recommended tolerance range, the probe light source window likely needs cleaning. Clean window according to instruction manual. When the observed reading is within the required tolerances, the instrument is fully calibrated.

Instrument Calibration Check:

1. Exit the exclusion zone and turn meter to "ON" position. Check that the meter is reading a value of zero.
2. Insert one end of T-tube into probe and other end into calibration gas. The third end of the T-tube should be attached to a flow meter.
3. Crack the valve on the calibration gas and read the value shown by the instrument. Record the value and calibration gas concentration on a field-data sheet.
4. If the value shown by the instrument is greater than $\pm 20\%$ of the calibration gas concentration, take meter outside of exclusion zone and recalibrate as outlined above.

Sample Measurement:

1. Place function switch in 0-20 ppm range for field monitoring. This will allow for most sensitive, quick response in detecting airborne contaminants.

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) outlines the procedures used in the development of wells. The purpose of well development is to remove well drilling fluids, solids, or other particulates that may have been introduced into the formation and/or well or deposited on the boring wall in a recently installed well during drilling and construction activities. This restores the hydraulic conductivity of the aquifer material surrounding the well to near pre-well installation conditions. Properly developed monitoring wells allow for the collection of ground water samples which are chemically and physically representative of the aquifer of concern. The procedure is also applicable to older or improperly developed wells that are suspected of not providing representative groundwater samples. Monitoring wells will be developed by pumping or bailing.

2.0 EQUIPMENT NEEDED

The following items are required to properly develop groundwater monitoring wells:

- Well keys
- Electronic water level indicator
- Calculator
- Field notebook
- Waterproof pen
- Submersible pump (or other appropriate pump), air-lift system or PVC, Teflon or stainless steel bailer (sized appropriately for well)
- Nylon rope or wireline (for deep wells) for bailing
- Surge block (sized appropriately for well)
- PVC or stainless steel pipe for operating surge block (sized appropriately for well)
- Thermometer
- pH meter (with automatic temperature compensation)
- Calibration pH buffer solutions
- Conductivity meter
- Polyethylene or glass container (for field parameter measurements)
- Plastic squeeze bottle filled with deionized water
- 5-gallon bucket
- Drums or other large container for development water, if required
- Appropriate health and safety equipment as specified in the Health and Safety Plan
- Appropriate decontamination equipment as specified in the decontamination SOP
- Well completion information

- Plastic sheeting, if required
- Generator or 12 volt battery if electric pump is to be used for developing

3.0 PROCEDURES

WELL DEVELOPMENT PROCEDURE

Well Development: URS personnel will direct a program for the development of the well without the use of dispersing agents, acids, or explosives. The objectives of well development are to: (a) assure that groundwater enters the well screen freely, thus yielding a representative groundwater sample and an accurate water level measurement; (b) remove all water that may have been introduced during drilling and well installation; (c) remove very fine-grained sediment in the filter pack and nearby formation so that groundwater samples are not highly turbid and so that silting of the well does not occur. Development will consist of surging and bailing or pumping until little or no sediment enters the well. Each monitoring well will be redeveloped until a minimum of five well volumes have been removed and pH, specific conductance, and temperature readings stabilize within 10% over a minimum of two successive readings.

If the addition of water is required to facilitate surging and bailing, only formation water from that well will be used. If this is not practical due to tightness of the formation then only bailing will be performed. In all cases, care will be taken not to collapse well screens during development activities.

The volume of water required for removal during development is calculated using the following method:

1. Measure the depth to water in the well from the measuring point. This is usually a notched point on the top of PVC riser pipe which has been surveyed.
2. Measure the total depth of the well from the same measuring point used for measuring the depth to water.
3. Calculate the height of water in the well casing by subtracting the depth of water from the total well depth.
4. Calculate the number of gallons of water corresponding to one well volume. This is done by multiplying the height of water in the well casing by the conversion factor corresponding to the inside diameter of the well casing.

The following equation shall be used to calculate the volume of water to be removed during well evacuation:

a) Volume of water in casing (gal) =
[ ]*water column (ft)*π*(well radius)^2*0.0043

b) Volume of water in annulus (gal) =

[ ]*water column (ft)*π*[(borehole radius)^2-(well radius)^2*0.0043*0.3

Multiply the volume of one well casing volume, plus the annulus volume if required, by [ ] to obtain the minimum volume of water to be evacuated.

During the well development activities, field measurements of temperature, pH, and specific conductivity are made, and the clarity, color, any presence of odors, and other comments regarding water quality are noted in the field notebook and on the well development log. The date, time, and volume of water removed is also recorded at this time. Measurements of pH, conductivity and temperature along with observations will be recorded as each well volume of water is removed. A sample of water will be collected for measurement of pH, conductivity, and temperature at the beginning of well development in order to establish a baseline for comparison with the water quality as well development proceeds.

3.0 DOCUMENTATION

Documentation of observations and data acquired in the field will provide information on well development and also provide a permanent record. These observations and data will be recorded with waterproof ink in a bound weatherproof field book with consecutively numbered pages and on the well development form attached.

As part of the development process, the following information will be recorded in the field book:

- Date
- Time
- Weather
- Personnel
- Well designation
- Well location
- Date(s) and time of well development
- Static water level from top of well casing before and after development
- Volume of water in well prior to development
- Volume of water removed and time of removal
- Depth from top of well casing to bottom of well
- Screen length
- Depth from top of well casing to top of sediment inside well, if present, before and after development

- Field measurements of pH, conductivity, and temperature taken during and after development
- Physical character of removed water throughout development (color, odor, and turbidity)
- Type and size/capacity of pump and/or bailer
- Description of development technique
- Decontamination observations
- Instrument calibration record

1.0 OBJECTIVE

This document defines the Standard Operating Procedure (SOP) for decontamination of equipment used in environmental sites.

2.0 EQUIPMENT NEEDED

The following equipment will be needed for decontamination procedures:

- Brushes
- Wash Tubs
- Buckets
- Scrapers, flat bladed
- Hot water – high pressure washer
- Paper towels
- Alconox detergent (or equivalent)
- Potable tap water
- Laboratory-grade deionized or distilled water
- Garden-type water sprayers

2.0 SAMPLING EQUIPMENT DECONTAMINATION PROCEDURES

Sampling equipment will be decontaminated at the sampling location under the following procedures:

- Personnel will wear the proper PPE to reduce the potential for exposure as required by the HASP.
- Partially fill two buckets with potable tap water, and add Alconox detergent to one of the buckets
- Use brushes to wash the sampling equipment (i.e. stainless steel bowls, stainless steel spoons, sampling utility knife, etc)
- Rinse sampling equipment in bucket containing potable tap water
- Rinse clean equipment with water sprayers containing distilled water (or equivalent)
- Place decontaminated equipment in clean area and allow to air dry.

3.0 DRILLING AND HEAVY EQUIPMENT DECONTAMINATION PROCEDURES

Drilling rigs will be decontaminated at a decontamination station located near a staging area. The decontamination station may be a temporary structure, or mobile trailer, capable of collecting all decontamination fluids. The following steps will be used to decontaminate drilling and heavy equipment.

- Personnel will suit up in proper PPE to reduce the potential for exposure as required by the HASP.
- Equipment showing gross impacted soil materials will be scrapped with a flat-bladed scrapper, and material containerized.
- Equipment that cannot be damaged by water, such as a drill rig, augers, drill bits, sampling equipment, shovels, etc, will be washed with a hot water, high-pressure sprayer, the rinsed with potable water.
- Following decontamination, drilling equipment will be placed on the clean drill rig and moved to the next sampling location. If equipment is not immediately used, it should be stored in a clean designated area.

Equipment rinsante samples of the decontaminated sampling equipment may be collected to verify the effectiveness of the decontamination procedures.

1.0 OBJECTIVE

This document defines the standard procedure for measuring water levels in wells. This Standard Operating Procedure (SOP) serves as a supplement to the sampling plan. This procedure describes equipment and field procedures necessary to collect water level measurements. The well locations and frequency of measurement are specified in the plan. This procedure is intended to be used together with the plan and other SOPs.

2.0 EQUIPMENT NEEDED

The equipment necessary to measure water levels includes:

- Solinst Model 101 water level meter or equivalent
- Two 5-gal buckets (with lids) or equivalent for decontamination
- Decontamination brushes
- Alconox soap
- Deionized or distilled water
- Potable water
- Spray bottle
- Field data sheets
- Field notebook
- Appropriate health and safety equipment

3.0 PROCEDURE

Appropriate health and safety equipment, as described in the Health and Safety Plan (HSP) should be worn during well opening, well measurement, and decontamination.

- The water level probe shall be decontaminated prior to use in each monitoring well.
- Observations concerning the well pad, surface or protective casing and other well conditions will be documented in the field notebook.
- The depth of the static water level and the total depth of the well will be measured using an electric water level meter. The measuring point for all the wells shall be the top of stainless steel monitoring well casing. If a reference mark is not found, then all well readings will be referenced to the north rim of the monitoring well riser pipe for standardization.
- The static water level and the total depth of the well shall be measured, recorded

on the water level data sheet, and then immediately rechecked.

- All columns of field data sheets shall be completed, including time of measurement. If measurements are taken over a several-day period, the date of each measurement should be clearly indicated on the form.
- Care shall be taken to verify the readings during each water level measurement period. Any significant changes in water level will be noted by comparing the most recent measurement with past measurements.
- After any measurement is taken, the water level probe shall be decontaminated.
- The water level indicator must be decontaminated before use, between wells, and at the conclusion of measurements.

3.2 DOCUMENTATION

Field data sheets or field notebooks will include date, time, well number, total well depth, water level, static water elevation, and comments. The data sheets or notebook shall be neat and legible, and shall be signed and dated by the person completing the page.

1.0 OBJECTIVE

This document defines the Standard Operating Procedure (SOP) and necessary equipment for collection of groundwater samples in monitoring wells, extraction wells, or piezometers using low-flow techniques. The term "low flow" refers to the velocity that the groundwater is removed from the soil formation immediately adjacent to the well screen.

In this technique, in order to withdraw water from within the well screen and to lessen drawdown, a pump that minimizes disturbance to the groundwater is operated at a low flow rate. The well is only purged within the screened interval until specific parameters have stabilized and as according to the site-specific work plan. Therefore, the groundwater samples collected are representative of the water bearing formation and hydraulically isolated from the water in the casing. The need to purge three well volumes, as required in traditional techniques, is not necessary with low flow purging and sampling. The low flow procedure described in this SOP is not necessarily applicable for every site or for wells screened in materials with very low permeability.

2.0 EQUIPMENT NEEDED

Equipment used during well purging and sampling:

- Well installation forms and boring logs for well being sampled
- Well keys
- Disposable latex or nitrile gloves
- Assorted tools (knife, screwdriver, etc.)
- New synthetic rope
- Pump and required accessories (described in more detail in following section)
- Electronic water level indicator with 0.01-foot increments
- Graduated cylinder
- Temperature meter
- pH meter (with automatic temperature compensation)
- Conductivity meter
- Turbidity meter
- Dissolved oxygen (DO) meter
- Oxidation reduction potential (ORP) meter
- Flow-through cell
- Calibration fluids

- Paper towels or Kimwipes
- Calculator
- Bound field logbook (logbook)
- Waterproof pen and permanent marker
- Plastic buckets
- 55-gallon drums or truck-mounted tank
- Plastic sheeting
- Appropriate decontamination equipment
- Cooler with ice
- Sample containers and labels
- Groundwater sampling form
- Chain-of-Custody form
- Appropriate health and safety equipment (e.g., photoionization detector (PID))

3.0 PROCEDURE

This section provides the step-by-step procedure for collecting groundwater samples in the field. Observations made during groundwater purging and sampling should be recorded in a logbook in accordance with the work plan.

A. Any equipment used in the sampling procedure that could contact groundwater should be properly decontaminated before each use.

B. Equipment should be calibrated based on the manufacturers' instructions. The frequency of calibration should be specified in the site-specific work plan. According to "Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures" (Unites States Environmental Protection Agency (USEPA), 1996), pH calibration should be performed with at least two buffers that bracket the expected range of values. Dissolved oxygen calibration must be corrected for local barometric pressure readings and elevation.

C. Before well purging begins, the following steps should be performed at each well:

- Inspect the well and surrounding site for security, damage, and evidence of tampering. If damage or tampering is evident, contact the project manager for guidance.
- Place clean plastic sheeting around the well (as necessary)

- Measure ambient volatile organic compounds (VOCs) background levels in the immediate vicinity of the well (i.e., using a PID or a flame ionization detector (FID) per the Health and Safety Plan (HASP).
- Remove the well cap and immediately measure VOCs at the rim of the well and record the readings in the logbook or on the groundwater sampling form. Give the water in the well adequate time to reach equilibrium.

D. After the well has reached equilibrium, the groundwater elevation should be measured to the nearest 1/100-foot. The total well depth and screened interval should be obtained from the well logs. Measuring the total depth prior to sampling should be avoided to prevent resuspension of settled solids in the well casings and to minimize the necessary purge time for turbidity equilibration. The total depth of the well should be confirmed after sampling has been completed.

E. Following measurement of the static groundwater elevation, the appropriate equipment will be slowly and carefully placed in the well. If the wells have light or dense non-aqueous-phase liquids (LNAPLs or DNAPLs) care should be taken to place sampling equipment below or above the NAPL.

Selection of the proper pump is important for low-flow sampling activities. USEPA guidance (1996) notes that dedicated sampling devices capable of purging and sampling are preferred over any other type of device. In addition, the pump must be capable of flow rates between 0.1 and 1.0 liter per minute. A variety of portable sampling devices are available, such as bladder pumps, electrical submersible pumps, gas-driven pumps, inertial lift foot-valve samplers (e.g. check-ball systems), and bailers (a list of pump manufacturers and suppliers is included on pg. 7).

When placing the equipment in the well, the pump intake should be set near the middle or slightly above the middle of the screened interval. If the screen length allows, the pump intake should be at least two feet from the bottom of the screen. Placing the pump intake near the top of the water column can cause stagnant water from the casing to be purged, but placing the pump intake near to the bottom of the well can cause mobilization and entrainment of settled solids from the bottom of the well.

F. Tubing should be connected from the pump to a flow-through cell. Then, calculate the volume of water to fill the flow-through cell. According to American Society for Testing and Materials (ASTM) Standard D 6771 (2002), the frequency of measurements should be equal

to the time required to completely evacuate one volume of the cell. This ensures that independent measurements are made.

G. The pump should be started at a low flow rate, approximately 100 mL/min or the lowest flow rate possible.

H. Water level measurements should continue every two minutes until the measurements indicate that significant drawdown is not occurring. According to ASTM standards (2002), allowable drawdown should never exceed the distance between the top of the well screen and the pump intake. Including a safety factor, also provided by ASTM, drawdown should actually not exceed 25% of this distance. This ensures that water stored in the casing is not purged or sampled. For example, for a 4-foot screen, the pump should be placed at the midpoint of the screen (two feet from the top of the screen to the pump intake). With a safety factor of 25%, this would require drawdown not to exceed six inches.

Once it has been established that significant drawdown is not occurring, the flow rate may be increased to </= 1 L/min (ASTM, 2002) or, if the flow rate remains the same, water level measurements need only to be taken periodically. However, when the flow rate is increased, water level measurements must continue every two minutes.

If drawdown surpasses 25% of the distance from the pump intake to the top of the screen even while pumping is occurring at the lowest flow rate possible, samplers should refer to the project specific criteria as found in the appropriate FSP or work plan.

I. Parameters should be documented on the groundwater sampling form and in the logbook. The time between parameter measurements is calculated as follows:

$T = \frac{V}{Q}$, where

T = time between measurements (minutes)

V = volume of the flow-through cell (liters)

Q = purge flow rate (liters per minute)

J. Sampling should occur as stated in the FSP or work plan. However, in most cases, purging will continue until specific parameters have stabilized over three consecutive flow-through cell volumes. **Table 1** provides guidelines that may be used for parameter stabilization as specified by ASTM Guidance on Low-Flow Purging and Sampling and Minimum-Purge Sampling. These guidelines are to be used in combination with professional judgment.

Table 1. Stabilization Guidelines for Low-Flow Sampling

Parameter	Stabilization Guidelines
	ASTM
DO	+/- 10% or +/-0.2 mg/L, whichever is greatest
ORP	+/- 20 mV
PH	+/- 0.2 units
Conductivity	+/- 3%
Temperature	Not Specified
Turbidity	Not Specified

K. After the relevant parameters have stabilized, the flow-through cell should be disconnected or bypassed for sampling. If, after a considerable number of readings have been taken, parameters have not stabilized, samplers should refer to the work plan or possibly use alternative sampling methods.

L. The flow rate should be adjusted to less than 0.5 L/min for sampling to minimize aeration during the sampling of volatiles.

M. A new pair of disposable latex or nitrile gloves should be put on immediately before sampling.

N. The constituents should be sampled for in the order given below:

- VOCs – Vials should be filled completely so that the water forms a convex meniscus then capped so that no air space exists in the vial. Turn the vial over and tap it to check for bubbles. If air bubbles are observed in the sample vial, remove the lid and attempt to fill the vial two more times, (being careful not to dump out any groundwater currently in the vial). If air bubbles are present twice more, discard the sample vial and repeat the procedure with a new vial. If, after three attempts, air bubbles are still in the vial, make a note of this and place the vial in the cooler.
- Gas sensitive parameters (e.g., ferrous iron, methane, alkalinity)
- Semivolatile organic compounds, pesticides, polychlorinated biphenyls, and herbicides
- Petroleum hydrocarbons
- Metals (unfiltered)
- Explosives
- Any filtered analytes (use in-line filters if possible)

O. Place all samples on ice inside a cooler immediately.

P. Each sample should be identified with the Sample ID, location, analysis number, preservatives, date and time of sampling event, and sampler.

Q. The sample time and constituents to be analyzed for should be recorded in the logbook and on the groundwater sampling form.

R. Chain-of-custody procedures should be started.

S. Sample equipment should be decontaminated.

T. The well sampling order should be dependent on expected levels of contamination in each well, if known, and should be determined prior to sampling. Sampling should progress from the least contaminated to the most contaminated well. Quality assurance/quality control (QA/QC) samples should be collected during groundwater sampling as required in the work plan and/or QAPP.

LIST OF SUPPLIERS WHO PROVIDE PUMPS SUITABLE FOR LOW-FLOW SAMPLING:

Field Environmental. 1-800-3930-4009. www.fieldenvironmental.com. Pumps: peristaltic, QED bladder pumps, Fultz rotor pump, control boxes, compressors, etc.

QED. 1-800-624-2026. www.micropurge.com. Pumps: bladder pumps, flow cell, compressors, etc.

Fultz Pumps. 1-717-248-2300. www.fultzpumps.com.

REFERENCES

ASTM 2002, Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations, ASTM D6771-02, American Society for Testing and Materials. West Conshohocken, PA.

Nielsen, David and Nielsen, Gillian. Technical Guidance on Low-Flow Purging and Sampling and Minimum-Purge Sampling. Second Edition. NEFS-TG001-02. April 2002.

USEPA. 1996. Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures. EPA/540/S-95/504. OSWER, April, 1996.

1.0 OBJECTIVE

This document defines the Standard Operating Procedures (SOP) for sample handling, documentation, tracking, and control and custody of environmental samples. This SOP serves as a supplement to the work plan.

2.0 EQUIPMENT NEEDED

The following equipment will be needed for sample control and custody procedures:

- Waterproof coolers (hard plastic or metal)
- Custody Seals
- Field forms such as a Chain of Custody (COC) or sample collection sheet
- Field Notebook
- Ice
- Sample Log-in Book
- Clear Tape
- Duct Tape
- Zip-Loc Bags
- Waterproof pens
- Permanent Markers.

3.0 PROCEDURES

Sample Identification

Samples collected during site activities shall have discrete sample identification numbers. These numbers are necessary to identify and track each of the many samples collected for analysis during the life of this project. In addition, the sample identification numbers will be used in the data base to identify and retrieve the analytical results received from the laboratory.

Each sample is identified by a unique code which indicates the site identification number, sample location number, sample matrix identifier, and sample depth.

The sample identification system will involve the following nomenclature "MWA-BBBB-CC" where:

"MWA" will denote

- MW#- Monitoring Well Location

"BBBB" will denote

- MMYY – Month and year of sampling quarter

  e.g.: First quarter (October), first year (2005), 1005

"CC" will denote QA/QC sample

- EB- equipment blank
- AD- analytical duplicate
- MS or MD – Matrix Spike or Matrix Duplicate
- TB- Trip Blank.

Sample Labeling

Sample labels will be filled out as completely as possible by a designated member of the sampling team prior to beginning field sampling activities each day. The date, time, sampler's signature, and the last field of the sample identification number should not be completed until the time of sample collection. All sample labels shall be filled out using waterproof ink. At a minimum, each label shall contain the following information:

- Project name and number,
- Sample number identification,
- Initials of sampler,
- Sampling location (if not already encoded in the sample number),
- Required analysis,
- Date and time of sample collection,
- Space for laboratory sample number, and
- Preservative used, if applicable.

Sample Handling and Shipping

The sample containers will be placed in reclosable Ziploc plastic storage bags and wrapped in protective packing material (bubble wrap). Samples will then be placed right side up in a cooler with ice (double bagged using plastic bags). Once the samples are collected, they must remain in the custody of the sampler or another worker from the site. The samples can also remain unattended in a locked vehicle so tampering with the samples will not be possible. Right before shipment, a custody seal should be placed over the opening of the cooler and then the cooler should be taped all the way around with clear packing tape to prevent tampering with the samples. Samples will be hand delivered or shipped by overnight express carrier for delivery to

the analytical laboratory. All samples must be shipped for laboratory receipt and analyses within specific holding times. This may require daily shipment of samples with short holding times. A chain-of-custody (COC) form will accompany each cooler. The temperature of all coolers will be measured upon receipt at the laboratory. A temperature blank will be included in each cooler for temperature measurement purposes.

Field Notes

Documentation of observations and data acquired in the field will provide information on the acquisition of samples and also provide a permanent record of field activities. The observations and data will be recorded using pens with permanent waterproof ink in a permanently bound weatherproof field log book containing consecutively numbered pages.

URS personnel will keep a bound field notebook while performing sampling and oversight activities on-site. Forms that will be used include: chain-of-custody, boring log, and soil sampling data sheets and field logbook. The field logbooks will record the following:

- Changes to the plan;
- Personnel conducting the site activities, their arrival and departure times and their destination at the site;
- Location where the work was performed;
- Date, time, weather conditions, equipment, and personnel on site;
- Daily information such as:
  - Work accomplished and the current site status,
  - Equipment calibrations, repairs and results, and
  - Site work zones;
- Specific work activities conducted such as
  - Work zone and headspace readings; and
- Incidents and unusual activities that occur on the site such as, but not limited to, accidents, breaches of security, injuries, equipment failures, or weather related problems.

The following sampling-related information will be recorded in the field logbook by the field sampling team:

- Sample number,
- Project identification,
- Sampling location,

- Required analysis,
- Date and time of sample collection,
- Type and matrix of sample,
- Sampling technique,
- Preservative used, if applicable,
- Sampling conditions,
- Observations,
- Initials of the sampler, and
- Samples collected.

Each page in the field books will be signed by the person making the entry at the end of the day, as well as on the bottom of each page. Anyone making entries in another person's field book will sign and date those entries. Any entry which is to be deleted will have a single cross out which is signed and dated. Sufficient information should be recorded to allow the sampling event to be reconstructed without relying on the sampler's memory. Photographic records will be developed through the use of digital photographs, showing pre-sampling and post-sampling conditions at each site.

Procedures to evaluate field data for this project primarily include checking for transcription errors on the part of field crew members and review of field notebooks. This task will be the responsibility of the URS Field Leader, who will otherwise not participate in making any of the field measurements or in adding notes, data, or other information to the notebook.

Sample Chain-of-Custody

During field sampling activities, traceability of the samples must be maintained from the time the samples are collected until the laboratory data is issued. Initial information concerning collection of the samples will be recorded in the field log book as described above. Information on the custody, transfer, handling, and shipping of samples will be recorded on a COC form.

The sampler will be responsible for initiating and filling out the COC form. The COC will be signed by the sampler or the field person responsible for sample handling when the sampler relinquishes the samples to anyone else. One COC form will be completed for each cooler of samples collected daily and if samples are not hand delivered, the COC will be placed in a Zip-Loc bag and shipped inside the cooler. COC forms will be used to document the transport and

receipt of samples from the field to the lab. Information required on a COC includes the following:

- Sampler signature/date/time/affiliation,
- Project number,
- Date and time of collection,
- Sample identification number,
- Sample type and matrix,
- Sample description/location,
- Analyses requested/comments,
- The total number of containers being sent to the lab and number of containers for each sample,
- The appropriate preservative used,
- If any samples are to be placed on hold at the laboratory, this should be clearly indicated on the COC in the comments section,
- Signature of person(s) relinquishing custody, dates, and times,
- Signature of person(s) accepting custody, dates, and times,
- Method of shipment, and
- Shipping air bill number.

The person responsible for delivery of the samples to the laboratory will sign the COC form, retain the last copy of the three-part COC form, document the method of shipment, and send the original and the second copy of the COC form with the samples. Upon receipt at the laboratory, the person receiving the samples will sign the COC form and return the second copy to the Project Manager. Copies of the COC forms documenting custody changes and all custody documentation will be received and kept in the central files. The original COC forms will remain with the samples until final disposition of the samples by the laboratory. The analytical laboratory will dispose of the samples in an appropriate manner 60 to 90 days after data reporting. After sample disposal, a copy of the original COC will be sent to the Project Manager by the analytical laboratory to be incorporated into the central files.

CHAIN OF CUSTODY RECORD

SHEET ___of___

URS CORPORATION

1001 HIGHLAND PLAZA DRIVE WEST, SUITE 300
ST. LOUIS, MISSOURI 63110
314-429-0100

PROJECT NO:	PROJECT NAME:	NO. OF CONTAINERS	CONTAINER DESCRIPTION / ANALYSES REQUESTED	REMARKS
SAMPLER'S: (Signature)				
DATE / TIME	SAMPLE I.D. NUMBER			

RELINQUISHED BY: (Signature)	DATE / TIME	RECEIVED BY: (Signature)	DATE / TIME
RELINQUISHED BY: (Signature)	DATE / TIME	RECEIVED AT LAB BY: (Signature)	DATE / TIME
METHOD OF SHIPMENT:		AIRBILL NO:	

1.0 OBJECTIVE

This document defines the Standard Operating Procedure (SOP) for sample handling, documentation, and tracking. This SOP serves as a supplement to the plan.

2.0 EQUIPMENT NEEDED

The following equipment will be required for this SOP:

- Waterproof coolers (hard plastic or metal)
- Custody Seals
- Field forms such as COC or sample collection sheet
- Field Notebook
- Ice
- Bubble Wrap
- Clear Tape
- Duct Tape
- Zip Loc Bags
- Sample Containers
- Waterproof Pen
- Permanent Marker.

3.0 SAMPLE CONTAINERS

Certified commercially clean sample containers will be obtained from the contract analytical laboratory. The lab will indicate the type of sample to be collected in each bottle type. The work plan lists the appropriate sample containers for the specific analyses require for each project.

4.0 SAMPLE PRESERVATION

Samples will be preserved at the time of the sample collection. Chemical preservatives, if necessary, will be added to the sample containers either by the laboratory prior to shipment to the field, or in the field by sampling personnel.

After sample collection, each container will be labeled and stored on ice at 4°C in an insulated cooler until packed for shipment until packed for shipment to the laboratory. The ice will be double bagged in Zip Loc storage bags. Freezing samples will not be permitted. Any breakable

sample bottles need to be wrapped in protective packing material (bubble wrap) to prevent breakage during shipping.

5.0 SAMPLE HOLD TIMES

Samples will be hand delivered or shipped by overnight express carrier for delivery to the analytical laboratory. All samples must be shipped for laboratory receipt and analyses within specific holding times. This may require daily shipment of samples with short holding times. The hold time varies for each type of analysis. It will be necessary to check with the lab to verify the hold times to determine how frequently samples need to be sent to the lab.

Documentation of observations and data acquired in the field will provide information on the acquisition of samples and also provide a permanent record of field activities. The observations and data will be recorded using pens with permanent waterproof ink in a permanently bound weatherproof field log book containing consecutively numbered pages.

Laboratory Standard Operating Procedures

VOLATILE COMPOUNDS BY GC/MS (EPA 8260B)

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Approved by: _____ Date: 3 April 2001

Title: Technical Manager, QA

STL X Savannah ___ Tallahassee ___ Mobile ___ Tampa West

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures that can be used to determine the concentration of volatile organic compounds (VOC) in water, wastewater, soils/sediments, wastes, oils, sludges, and solids. The attached quantitation report lists the target compounds, an example of the retention time order of each target compound, the quantitation and confirmation ions of the target compounds, and internal standard assignments.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision criteria for each target compound are listed in Section 5 of the current revisions of the STL Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan.*

2.0 SUMMARY OF METHOD

2.1 Volatile organic compounds (VOC) are purged from the sample matrix with helium. The VOC are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOC are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOC, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.2 Aqueous samples may be purged at ambient conditions (recommended) or at 40C (optional). Five to twenty-five milliliter aliquots of the sample may be purged. The calibration standards and the associated QC must be analyzed under the same conditions and volume.

2.3 Low-level (nominally <1mg/kg) soil samples are purged at 40C in a purge and trap instrument designed to add water and internal standards to the vial containing the sample without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards are purged under the same conditions.

2.4 High level soils (nominally >1mg/kg) and waste samples are extracted with methanol-1mL of methanol per gram of sample. An aliquot of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at ambient temperature using the same instrument conditions and calibration used for aqueous samples.

2.5 This method is based on the guidance in SW-846 Methods 8260B and 5035.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedure that you do not understand or that will put yourself or others in a potentially hazardous situation.

3.2 Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, eye protection, or other equipment should be used. Standards and highly contaminated samples should be handled in a hood.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to safely handle these materials.

3.4 The exit vent of the split injector must have a carbon trap in-line to collect the volatile compounds that are vented during the injection of the sample. The traps should be changed a minimum of every three months and disposed of in accordance with STL-SL SOP CA70: *Waste Management.*

4.0 INTERFERENCES

4.1 VOCs commonly used in the laboratory are potential sources of contamination. Methylene chloride, acetone, Freon-113, MEK, hexane, toluene, and isopropanol are used in the laboratory and tend to present the most problems.

4.2 The volatiles lab must be kept as free from contamination as possible. Highly contaminated samples must be segregated from routine samples. Contact with sections of the laboratory where solvents are used should be minimized. Refrigerator blanks should be prepared, stored, and analyzed to evaluate the sample storage areas for possible contamination. Guidance is provided in STL-SL SOP AN70: *Segregation of Low and High Concentration Volatile and Semivolatile Samples.*

4.3 Matrix interferences may be overcome by the use of the secondary ions for quantitation. An example of this is the use of mass 82 for quantitation with chlorobenzene-d5 internal standard when a potential co-eluter, 1,1,1,2-terachloroethane, is a target compound. One of the mass fragments of 1,1,1,2-tetrachloroethane is mass 117, which is the recommended quantitation ion for chlorobenzene-d5. The use of the secondary ions should be used for quantitation in such cases when the lab can clearly demonstrate matrix problems. Mass 58 is recommended for quantitation of acetone due to the elution of a hydrocarbon at the same retention time.

4.4 The analysis of highly contaminated samples (>1mg/L or >1mg/kg) can affect succeeding analyses. Carry-over can occur when low concentration samples are analyzed after high concentration samples. Trap replacement and purging of the entire purging system may be necessary when carry-over is suspected. Reagent blanks must be analyzed when carryover is suspected to demonstrate that the system is free from contamination.

4.5 The Teflon seals of the purge and trap device can absorb and outgas many of the compounds that are included in this method. These Teflon fittings should be periodically checked for integrity. If contamination of the fittings is suspected, the fittings may be heated at 105 C for one hour or replaced.

5.0 SAMPLE COLLECTION, HANDLING, AND PRESERVATION

5.1 Liquid samples are collected with no headspace in 40mL vials equipped with Teflon-lined caps. The samples are acidified at the time of collection with about 0.10mL of concentrated HCl per 40mL of sample. The acid prevents the biological degradation of the aromatic compounds and prevents the dehydrohalogenation of some of the chlorinated alkanes. The sample must be iced at the time of collection and refrigerated at 4C (less than 6C with no frozen samples) in the lab until analysis.

Check each sample vial at the time of receipt for the presence of "bubbles". If the bubbles are less than 3mm in diameter, the vial is acceptable. If the bubble is greater than 3mm, use another vial. Notify the department supervisor or project manager if there are no acceptable vials for analysis.

A "sacrificial" vial or the vial used for screening analysis is used to check the sample pH. If the sample pH is greater than 2, notify the department supervisor or project manager. If directed by supervisor or project manager, hydrochloric acid may be added through the septum to bring the pH <2. Do not add more than 400uL (0.40mL) of 1:1 HCl to a VOC vial. If pH cannot be adjusted to <=2 without destroying the integrity of the sample, the sample must be analyzed within 7 days of collection.

The holding time for samples preserved with HCl is 14 days for all target compounds. The holding time for un-preserved samples is 7 days.

5.2 Soils: Soils are routinely collected in duplicate in Encore samplers. A "bulk" sample is also routinely collected in a 125-mL jar fitted with a Teflon-lined cap. The bulk sample can be used for the methanol extraction if the concentration of the sample collected in the Encore exceeds the working range of the analytical system.

Soils collected in Encore samplers must be analyzed within 48 hours of collection or must be transferred within 48 hours of collection to sealed vials containing sodium bisulfate solution or methanol. If the sample contains high levels of carbonates, the sample is preserved with water and frozen until the time of analysis. The procedure for preparing soil samples is given in Section 9.2.

The hold time of the preserved sample is 14 days from the date of collection. The hold time for frozen samples is 14 days from the date of collection.

5.3 High level soil and waste samples are collected in glass containers (usually 125-mL clear glass) equipped with Teflon-lined caps. Soil samples may also be submitted as core samples contained in Encore samplers, metal or plastic "tubes", or in 40-mL VOA vials. The samples are iced at the time of collection and stored at 4C (less than 6C with no frozen samples). The holding time for soil and waste samples subjected to methanol extraction is14 days from date of collection; that is, the extraction and analysis must be completed within 14 days of collection.

5.4 TCLP leachate samples are collected with no headspace in Tedlar bags or syringes. The leachate samples are acidified at the time of collection (after the leaching procedure) with about 0.10mL of concentrated HCl per 40mL of sample and stored at 4C (less than 6C with no frozen samples) from the time leaching is completed until the analysis. The acidified leachate sample must be analyzed within 14 days of the leaching procedure. If the sample is not acidified, the leachate must be analyzed within 7 days of the leaching procedure.

NOTE: Samples that are suspected of having very high concentrations of VOC should be segregated from the "routine" samples and stored in a manner that will minimize sample and laboratory contamination. See STL-SL SOP AN70. If possible, keep the field QC in the same storage refrigerator as the samples.

6.0 APPARATUS AND MATERIALS

The apparatus and materials listed in this section may vary from lab to lab. The items listed are to give guidance and to provide a general overview of the equipment employed in this analysis.

6.1 Mass spectrometer: equipped with a capillary direct interface and a split/splitless injector or molecular jet separator

6.2 Gas chromatograph, compatible with the MS and purge and trap systems. If the GC is equipped with an injector that is operated in the split mode, the exit vent must have a carbon trap in-line to collect the volatile compounds that are vented during the transfer from the purge and trap device. The carbon traps should be changed a minimum of every three months.

6.3 Purge and trap device Tekmar 3000 Liquid Concentrator or equivalent

6.4 Supelco Vocarb 3000 trap or equivalent, Other traps may be used as long as the target compounds can be detected at the required quantitation limit.

6.5 Archon soil analyzer for low level soils, compatible with Tekmar purge and trap instruments. The instrument must be capable of automatically adding water and internal standard to the container while maintaining the septum seal, heating the sample to 40C, and spinning the stir bar to mix the sample during the purging step.

6.5 Data System compatible with the analytical system

6.6 Microsyringes: 10ul, 25ul, 50ul, 100ul, 250ul, 500ul, 2.5mL

6.7 Gastight syringe: 5mL, 25mL with luerlock tip

6.8 Volumetric flasks: 1.0mL, 10mL, 100mL

6.9 Recommended Columns

J&W DB-624: 60m x 0.32mm ID, 1.8um film
J&W DB-624: 20m x 0.18mm ID, 1.8um film

7.0 REAGENTS

Reagents must be tracked in accordance with STL-SL SOP AN44: *Reagent Traceability.*

7.1 Reagent water-free of volatile contaminants (obtained by purging with inert gas or carbon filtration)

7.2 Methanol-Burdich and Jackson, Purge and Trap grade

7.3 Sodium bisulfate-reagent grade. This salt is hydroscopic and should be stored in a dessicator.

7.4 Soil preservation solution- Slowly add, while stirring, 200g of sodium bisulfate to a 1.0-L volumetric containing about 700mL of reagent water. After the salt has dissolved, dilute to volume with reagent water, transfer to a storage container, and store the solution in an area free from VOC-especially water-soluble solvents such as acetone. The reagent should be tested prior to use by the analysis of a blank containing 5mL of the solution. The reagent is acceptable if it meets the same criteria as a method blank.

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions purchased from vendors or from stock standards prepared from neat materials. Certificates of analysis or purity must be received with all stock solutions or neat compounds. All preparation steps must be in accordance with STL-SL SOP AN41: *Standard Material Traceability.*

8.1 Preparation of **Stock Standards** from Neat Compounds

The lab should attempt to obtain a certified primary standard or secondary standard before preparing stock standards from neat materials. If primary stock standards must be prepared in-house, the target concentration range is from 2000ug/mL to 10000ug/mL. SL-SOP AN43: *Standard Preparation* gives the general instructions for the preparation of the stock solutions from neat materials.

8.2 Preparation of the **Working Standard** from Stock Standards

The working standard is prepared from the primary stock standards that are either prepared from neat compounds or purchased as certified solutions. The working standard contains one or more of the target compounds at a concentration suitable for preparing the calibration standards, generally 10-200ug/mL. A known volume of the working standard is then added to a known volume of reagent water to make the calibration standard.

The standards and standard concentrations listed in Table 1 are the suggested for routine use. If other "recipes" are used, the lab must document the standard preparation procedures in the standard traceability log.

8.3 Preparation of the **Calibration Standards** from the Working Standards

The calibration standards are the standards that are analyzed on the instrument. The calibration standard is made by adding a known volume of the working standard to a known volume of reagent water. The instrument must be calibrated using a minimum of five calibration standards. The lowest level standard must be at the reporting limit and the rest of the standards will define the working range of the analytical system.

8.3.1 Add 5.0mL of reagent water to a 5mL-glass syringe or 25ml of reagent water to a 25-ml glass syringe.

8.3.2 Add a known volume of the working standard to 5.0mL or 25ml of reagent water.

NOTE: The calibration standards for the low level soils are prepared using the same procedures as for the 5mL water purge except that the standards are purged at 40C. The lab has the option of using blank sand in the calibration standards.

The calibration standards listed in Table 1 are the suggested for routine use. If other "recipes" are used, the lab must document these standard preparation procedures in the standard traceablity log. A 5mL-purge volume may be used for low level (nominal RL of 1ug/L) if the instrument has sufficient sensitivity to detect the targets and the calibration criteria is met.

9.0 SAMPLE PREPARATION

Composite samples can be prepared using the guidance provided in STL-SL-SOP AN70.

9.1 **Aqueous samples** are analyzed directly by purge and trap/GC-MS. No sample preparation is necessary except to homogenize the sample prior to subsampling. The pH of liquid samples is checked and recorded prior to analysis to determine if the sample has been properly preserved.

9.2 Preparation of Soil Samples (5035)

9.2.1 Remove the Encore samples and the bulk sample from the storage area.

9.2.2 Test an aliquot of the bulk sample for the presence of carbonates.

- Transfer 5g of sample from the bulk sample to a 40mL vial..
- Add 5ml of the sodium bisulfate solution and shake the vial .
- If the sample exhibits effervescence, the Encore samples should be preserved as described above using 5mL of volatile-free water in place of the sodium bisulfate solution and placed in a freezer at –10C. The analytical hold time for frozen samples is 14 days from collection.
- If no effervescence is noted, the Encore samples may be preserved with 5mL soil preservation solution.

9.2.3 Add a stir bar to a vial and weigh the vial and record its tare weight(or tare the vial and stir bar weight by pressing the autotare button).

9.2.4 Transfer the sample from the Encore sampler to the tared vial and record the weight of the sample log.

If the sample effervesced during the carbonate test (9.2.2), add 5.0mL of reagent water and freeze at –10C. The hold time is 14 days from collection.

If not, add 5.0mL of the soil preservation solution, seal the vial, and store the sample at 4C until the time of analysis. The preserved sample must be analyzed within 14 days of collection.

NOTE: A preparation blank is prepared when Encore samples are transferred. The preparation blank contains the same reagents as the samples-either 5mL of reagent water or 5mL of soil preservation solution.

9.3 A methanol extraction is prepared when the concentration of the target compounds (by direct purge) exceeds the working range of the calibration curve. The bulk sample, collected in the 125-mL sample container, can be used to prepare the methanol extraction. Carry out the preparation quickly to minimize the loss of volatiles.

-Mix the sample with a stainless steel spatula and transfer 10g (+/- 0.5g) to a glass vial.

-Add 8uL of the surrogate spiking solution (2500ug/mL) to the sample and quickly add 10mL of purge and trap grade methanol. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 10g and 100% percent solids, is calculated:

$$Ct(ug/kg,dw) = \frac{0.008mL \otimes 2500ug/mL}{0.010g \otimes solids} = 2000ug/kg,dw$$

-Shake the sample for two minutes. Allow the solvent to separate from the solids portion of the sample and transfer a 1-2mL aliquot of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

-For each batch of twenty or fewer samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding 8uL of the surrogate spiking solution to 10mL of purge and trap grade methanol. Assume a sample weight of 10g. Analyze 125uL of the extract.

The lab control standard is prepared by adding 8uL of the surrogate spiking solution and 8uL of the matrix spiking solution to 10mL of purge and trap grade methanol. Assume a sample weight of 10g. Analyze 125uL of the extract.

The matrix spikes are prepared by adding 8uL of the surrogate spiking solution (2500ug/mL) and 8uL of the matrix spiking solution (2500ug/mL) to 10-g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for two minutes. Analyze 125uL of the extract or a smaller volume if the VOC concentration is high.

-Add 125uL of the extract (or a smaller volume if the VOC concentration exceeds the linear range of the system with 125uL) to 5.0mL of water (or to 25mL if the calibration is based on 25mL). Add the internal standard solution and analyze the sample using the ambient water calibration.

9.4 Methanol Extraction for Wastes

Carry out the preparation quickly to minimize the loss of volatiles.

9.4.1 Mix the sample with a stainless steel spatula and transfer 1g (+/- 0.2g) to a glass vial.

9.4.2 Add 10uL of the surrogate spiking solution (2500ug/mL) to the sample and quickly add 10mL of purge and trap grade methanol. If the sample is completely soluble in the methanol, dilute to a final volume of 10mL. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 1.0g , is calculated:

$$Ct(ug/kg) = \frac{0.010mL \otimes 2500ug/mL}{0.0010g \otimes solids} = 25000ug/kg$$

9.4.2 Shake the sample for one minute. Allow the solvent to separate from the solids portion of the sample and transfer 1mL to 2mL of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

For each batch of twenty or fewer samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding 8uL of the surrogate spiking solution (2500ug/mL) to 10mL of purge and trap grade methanol. Assume a sample weight of 1.0g. Analyze 100uL of the extract.

The lab control standard is prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) and 10uL of the matrix spiking solution (2500ug/mL) to 5.0mL of purge and trap grade methanol. Assume a sample weight of 5.0g. Analyze 100uL of the extract.

The matrix spikes are prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) and 10uL of the matrix spiking solution (2500ug/mL) to 1g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for one minute.

Add 100uL of the extract (or a smaller volume) to 5.0mL of water (or to 25mL if the calibration is based on 25mL). Add the internal standard solution and analyze the sample using the ambient water calibration.

NOTE: Waste samples may require significant dilution prior to analysis.

10.0 PROCEDURE

The following instrument conditions are recommended. The actual conditions may vary due to differences in instrumentation. The lab must document the instrument conditions in the maintenance log, the data system, or on the analysis log.

10.1 Instrument Conditions

10.1.1 GC Conditions
GC conditions may vary according to the environment and condition of each instrument. The lab must document the instrument conditions to assure consistent results and to aid in trouble-shooting the analytical system. Each lab is responsible for assuring that the conditions necessary to achieve adequate separation and sensitivity of the target analytes are maintained.

10.1.1.1 Example GC temperature program

Initial column temperature: 35 C for 3 minutes
Column temperature program 1: 20C per minute
Intermediate column temperature: 70C for 4 minutes
Column temperature program 2: 10C per minute
Final column temperature: 200C for 5.25 minutes

10.1.1.2 Column flow: Approximately 5-10mL/minute helium with a make-up of 20-25mL/minute helium. Total flow into the jet separator should be about 30mL/minute. The vacuum gauge on the jet separator will read about 0.5Torr.

If no jet separator is used and the column is plumbed directly into the source, the column flow should be adjusted to 0.5-1.0ml/min and a split ratio (desorb to column flow) of about 40:1 established. Smaller bore capillary columns (0.18 to 0.32mm) are required if the column is plumbed directly into the source

10.1.1.3 Mass Spectrometer and interface parameters

Jet separator temperature: 240C
Mass spectrometer interface: 240C
Mass spectrometer source temperature: factory set at 300C
range: 35-300amu, with a minimum scan cycle of 1 scan per second

10.1.2 Purge and Trap Conditions
The purge and trap conditions listed in this section are for guidance. The lab must document the actual conditions used. The purge time must be 11 minutes. Other parameters may be varied to optimize the detection of the target compounds.

10.1.2.1 "Three ring trap"-charcoal, Tenax, silica gel
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 180C
Bake time: 8 minutes at 225C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

10.1.2.1 VOCARB 3000 trap
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 225C
Bake time: 8 minutes at 250C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

The purge flow must be balanced for adequate sensitivity of the target compounds. If the purge flow is too high, the response of the gases will be low and not reproducible. The SPCC criteria for chloromethane may not be achieved if the purge flow is too high. If the purge flow is too low, the response of the more water-soluble targets-ketones, ethers, bromoform-may be low and the reporting limit may not be achieved on a routine basis.

10.2 BFB Tune Check

10.2.1 Fifty nanograms of 4-BFB must be analyzed at the beginning of each 12-hour clock as a check on the **"tune"** of the mass spectrometer. Meeting the tuning criteria ensures that the instrument is measuring the proper masses in the proper ratios. The 4-BFB analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of 4-BFB. All other instrument conditions must be identical-the mass range, scan rate, and multiplier voltage. If the instrument is configured for direct injection, 50ng of 4-BFB may be injected directly on to the column. If the purge and trap is used to analyze the 4-BFB, the purge and trap conditions must be the same as for the calibration standards and samples.

10.2.2 Evaluation of the 4-BFB peak.

10.2.2.1 The chromatogram should exhibit acceptable baseline behavior and the 4-BFB peak should be symmetrical. A spectrum of the baseline that shows high abundances of mass 40 (Argon) and mass 44 (carbon dioxide) may indicate a leak or contaminated carrier gas.

10.2.2.2 The spectrum of the 4-BFB must meet the criteria listed in the attached SOP Summary. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans +/- 5 scans from the apex can be evaluated for the 4-BFB criteria. Consecutive scans within this range can be averaged to meet the criteria.

10.2.2.3 The following records must be kept for each 4-BFB analysis that meets the criteria:
- the date, time, and data file of the analysis
- a spectrum of the scan or averaged scans
- a tabulation of the ion abundances of the scan

10.2.2.4 The 4-BFB analysis should be evaluated as to the relative size of the 4-BFB peak under the m/z 95 profile. A benchmark area window should be established for each instrument. Response outside of this window suggests instrumental problems such as a poor purge, clogged jet separator, leak in the Tekmar purging device, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open , or other anomalies.

10.2.2.5 If the 4-BFB fails to meet the acceptance criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the 4-BFB analysis, other corrective measures may include remaking the 4-BFB standard and/or cleaning the mass spectrometer source.

10.3 Initial Calibration

After the 4-BFB criteria has been met, the initial calibration standards are analyzed. Prepare the initial calibration standards according to the example recipes in the SOP appendices or lab-specific recipe. The lab must document the "recipe" used to prepare the calibration standards. The lowest level calibration standard must be at or below the routine RL and the other calibration standards will define the working range of the system.

10.3.1 Remove the plunger from the syringe and fill the barrel to overflowing with reagent water (syringe valve in the "red" position).

10.3.2 Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume(5mL or 25mL)

10.3.3 Briefly remove the syringe valve and inject the standards and internal standards into the syringe.

NOTE: Use the internal standard (IST) mix when preparing the calibration standards for analysis. The surrogates are already included in the standard mixes.

10.3.4 Load the standard(s) onto the purge and trap device and begin the analysis. All pertinent information concerning the standards must be recorded on the analysis log. The standards must be clearly identified and traceable to the preparation steps.

NOTE: The standards for low-level soil samples are prepared in the same manner as the 5mL standards. The standards for the low-level soils are purged at 40C. The lab has the option of using blank sand or soil in the calibration standards and the blank in the low level soil analysis.

10.3.5 After the acquisition has taken place, evaluate the calibration standards to ensure that each target compound, surrogate, and internal standard has been correctly identified. The analyst must be careful to complete this step before proceeding.

10.3.6 After each target compound, surrogate, and internal standard has been correctly identified, the relative response factor for each target compound and surrogate is calculated using the data system or using a PC spreadsheet as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where
Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (see the attached quantitation report for a list of the compounds that are associated with the various internal standards)
Cx = concentration or mass on-column of the target compound being measured (ug/L or ug/kg OR ng or ug on-column)
Cis = concentration or mass on-column of the internal standard (ug/L or ug/kg OR ng or ug on-column)

The average relative response factor (RRFavg) is calculated for each target compound and each surrogate compound:

$$RRFavg = \frac{RRF1 + RRF2 + ... + RRFn}{n}$$

where n = number of calibration levels

Calculate the standard deviation (SD) for the target compounds and surrogates at all calibration levels:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RFi - RFavg)^2}{n-1}}$$

where
Rfi = response factor of a target compound in the individual calibration level
Rfavg = average response factor
n= number of calibration levels

10.3.7 Calculate the relative standard deviation (% RSD) of the calibration levels for each target:

$$\%\,RSD = \frac{standard\ deviation}{RRFavg} \otimes 100$$

10.3.8 The results of the initial calibration are evaluated against the Calibration Check Compound (CCC) criteria and the System Performance Check Compound (SPCC) criteria, which are listed below. The CCC and SPCC criteria must be met before samples can be analyzed.

Calibration Check Compounds – CCC Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	Continuing Calibration
<=30% RSD	<=20% difference from initial calibration

System Performance Check Compounds-SPCC

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30 (0.10 for 25mL purge volume)

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

10.3.9 After the initial calibration criteria (CCC and SPCC) have been met, each target is evaluated for linearity.

If the %RSD of the target compound is less than or equal to 15%, the average response factor can be used for quantitation of samples.

If the %RSD of the target compound is greater than 15%, a regression curve (linear, quadratic, etc) must be used for the quantitation of samples. A regression curve may also be used for the compounds that have %RSD less than 15%. The results can be used to plot a calibration curve of response ratios-Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration or mass on-column of the target compound being measured (ug/L or ug/kg OR ng or ug)
Cis = concentration of the internal standard (ug/L or ug/kg OR ng or ug)

If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples.. Regression curves may be forced through zero but it is recommended that the curve be evaluated without forcing through zero first and then with the curve forced through the origin. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range

When more calibration levels are analyzed than required, individual compounds may be eliminated from the lowest or highest calibration levels(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of the calibration curve be eliminated without eliminating the entire level.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

8000B exception: evaluation of the "grand mean": If the average %RSD of ALL (all targets including CCC and SPCC) compounds in the initial calibration is less than 15%, the average response factor can be used for quantitation of all target compounds. The recommended course is to use regression curves, as described above, to quantify targets where the %RSD criterion (<=15%) is exceeded.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

10.3.10 After the initial calibration criteria has been met, the method blank is analyzed. 5.0mL or 25mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in Table 5 of the STL-SL CQAP or other specified QAP.

If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compounds must meet the criteria.

If the concentration of any target compound is above the RL in Table 5 of the STL-SL CQAP, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of samples can take place.

If the method blank repeatedly fails to meet the criteria, contact the immediate supervisor to determine the cause of the problem and to determine a course of action. This action may include re-cleaning the sparging tubes (with soap, hot water, and methanol), purging the effected autosampler ports with heated methanol, flushing the purge and trap ALS concentrator with methanol, replacing the trap, changing the transfer line, and changing the column. A method blank is then analyzed after taking the corrective action to demonstrate that the contamination has been eliminated. Once the system is determined to be free from contamination, sample analysis may begin. Method blanks may be required after the analysis of samples that contain very high levels of VOC.

10.4 Continuing Calibration Verification

At the beginning of each 12-hour clock, the tune of the instrument must be checked by the analysis of 50ng of 4-BFB. This criteria must be met before the analysis of the calibration check standards can take place.

10.4.1 After the tune criteria has been met, a continuing calibration check standard(s) is analyzed. The continuing calibration standard should be at a nominal concentration of 50ug/L-kg for 5ml/5g samples and 10ug/L for 25mL with ketones and poor purgeables at higher concentrations. The CCC and SPCC criteria (Section 10.3.8) must be met before the analysis of the method blank and samples can take place. The percent difference (%D) is calculated as follows:

$$\%D = \frac{RRFavg - RRFccv}{RRFavg} \otimes 100$$

where
RRFavg = average response factor from initial calibration
RRFccv = response factor from the check (12-hour) standard-calibration verification

The percent drift (%Drift) may also be used to evaluate the change/deviation of the curve:

$$\%Drift = \frac{Ci - Cccv}{Ci} \otimes 100$$

where
Ci = Calibration Check Compound standard concentration
Cccv = measured concentration using the selected quantitation method

NOTE: The SPCC criteria (10.3.8) must be met even if the regression curve option is used for quantitation. If this criteria is not met, corrective action must be taken. The corrective action may include reanalysis of the calibration check standard or preparation of a new secondary stock standard and reanalysis of the calibration check standard. If subsequent analysis of the standard is still out of criteria, a new initial calibration curve must be analyzed and evaluated.

10.4.2 The calibration standard (CCV) must also be evaluated for internal standard retention time and response.

If the retention time of any internal standard changes by more than 30 seconds from the retention times of the internal standards in the initial calibration, the analytical system must be inspected for problems and corrective action instituted.

If the extracted ion current profile (EICP) area for any of the internal standards changes by more than a factor of two (-50% to +100%) from the last calibration check standard, the analytical system must be inspected for problems and corrective action instituted. If the CCV is the first one after the initial calibration, compare the ISTD response to the corresponding level in the ICAL.

10.4.3 After the continuing calibration criteria has been met, the method blank is analyzed. 5.0mL or 25mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in Table 5 of the STL-SL CQAP.

If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compound must meet the criteria.

If the concentration of any target compound is above the RL in Table 5 of the STL-SL CQAP, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of client samples can take place.

10.5 Aqueous Sample Analysis-5.0mL to 25mL

The analyst must use the same volume as was used for the calibration standards-if a 5mL sample is used, it must be quanted off of the 5mL calibration curve; if a 25ml sample is used, it must be quanted off of the 25mL calibration curve. Samples are analyzed only after the tune criteria, the calibration (initial or continuing) criteria has been met, and the method blank criteria has been met. See the SOP Summary for the analytical sequence.

10.5.1 Remove the samples to be analyzed from the refrigerator and allow the samples to come to ambient temperature.

10.5.2 Put on a pair of gloves before transferring the sample from the vial to the syringe. The sample is most likely preserved with acid or may contain toxic or hazardous chemicals or biologically active components that may cause skin irritations. ***Gloves must be worn when handling samples.***

10.5.3 Mix the contents of the vial by inverting the vial several times. Check to see if there are air bubbles present in the sample. If air bubbles are present, use another vial if available. Make a note on the analysis log if the sample used contained bubbles and notify the supervisor and/or the project manager.

10.5.5 Remove the plunger from the glass syringe. Attach a syringe valve to the syringe Luer-tip to prevent sample from spilling out of the syringe when sample is added.

10.5.5 Open the vial of the well-mixed sample and gently pour the sample into the syringe barrel. The sample should fill the barrel of the syringe and overflow to allow trapped air bubbles to escape.

10.5.6 Replace the plunger into the syringe barrel. Try not to let air bubbles get into the barrel. If air bubbles are present, turn the syringe up, open the syringe valve , and expel the air while adjusting the volume to 5.0mL or 25mL. If no air bubbles were trapped, adjust the syringe to volume.

NOTE: For TCLP leachate samples, use 1.25mL of sample (1:4 dilution).

10.5.7 Open the syringe valve and inject the internal standard/surrogate (ISSU) mix into the sample.

10.5.8 Transfer the sample from the syringe to the purge and trap device. Record all of the sample identification information on the analysis log. Check the pH of the sample with pH paper and record the pH on the instrument log or other appropriate log.

10.5.9 Analyze the samples using the purge and trap and GC/MS conditions used for the initial and continuing calibration standards.

10.5.10 Determine the concentration of the samples and QC items. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

NOTE: Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client. For TCLP analyses, every reasonable effort should be made to achieve the regulatory level with out instrument overload.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with t the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower RLs.

A dilution is made when a volume of the sample is mixed with the reagent water to a final volume of 5.0mL or 25ml,depending on which curve is being used. The dilution factor is calculated by dividing the volume of sample into the volume used for the calibration curve.

$$DF = \frac{final\ volume\ of\ dilution(mL)}{volume\ of\ sample\ used(mL)}$$

For example, if 1.0mL of sample is diluted to final volume of 5.0mL, the dilution factor is 5. (5.0/1.0 = 5). If 1.0mL of sample is diluted to a final volume of 25mL, the dilution factor is 25 (25/1=25).

The following table gives some dilution factors:

Volume of Sample (mL)	Volume of Reagent Water (mL)	Final Volume (mL)	Dilution factor
5.0	0	5.0	1
2.5	2.5	5.0	2
1.0	4.0	5.0	5
0.5	4.5	5.0	10
0.10	4.9	5.0	50
25.0	0	25.0	1
5.0	20.0	25.0	5
2.5	22.5	25.0	10
1.0	24.0	25.0	25
0.50	24.5	25.0	50
0.10	24.9	25.0	250

NOTE: The same volume of internal standard/surrogate mix (ISSU) is added to the dilution as was added to the undiluted sample.

10.6 Low Level Soil Samples by Heated Purge and Trap (Method 5035)

The soil analytical system is calibrated using the same concentrations as the 5mL purge. The tune, initial and continuing calibration criteria, and the method blank criteria must be met before samples are analyzed. Standards and QC items must be analyzed under the same heated purge and trap conditions.

Remove the samples to be analyzed (Section 9.2) from the refrigerator or freezer and allow the sample to come to ambient temperature. Inspect the vial for cracks or obvious breaches in the septum. Load the samples on to the soil-purging unit and analyze according to the sequence described in Appendix B.

Liquid field QC for soils (trip blank, field blank, etc.) should be analyzed with the associated soil samples, using the same preparation and analytical procedures, including the heated purge. Report the results for liquid trip blanks as ug/L.

10.7 Analysis of Methanol Extracts of Soils and Wastes

The methanol extraction is used when the concentration of one or more target compounds exceeds the linear range of the low-level purge technique (>1000ug/kg). or if the concentration of VOC in the soil or waste samples is high. Samples are analyzed only after the 4-BFB criteria, the calibration criteria (initial and continuing), and the method blank criteria has been met. Medium level soil extracts are quanted using the ambient purge calibration curve. Sample preparation steps are included in Section 9.

10.7.1 Remove the plunger from the 5.0-mL syringe and fill the barrel to overflowing with reagent water(syringe valve in the "red" position). Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume(5mL)

10.7.2 Briefly remove the syringe valve and inject the sample extract and 5uL of the internal standard (IST) solution into the syringe. Use 125ul of the extract for soils and 100uL of the extract for wastes. Smaller aliquots are used if the concentration of target analytes exceed the working range of the system.

NOTE: Use the internal standard (IST) mix when preparing the medium level samples. Recall that the surrogates have already been added to the sample during the methanol extraction step (Section 9).

10.7.3 Load the sample on to the purge and trap device and begin the analysis. All pertinent information concerning the samples must be recorded on the analysis log. The samples must be clearly identified and traceable to the extraction log. These conditions must be the same as was used for the initial and continuing calibration standards-ambient purge for aqueous samples.

10.7.4 Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, a smaller aliquot of the methanol extract is reanalyzed to bring the highest target within the upper half of the calibration curve. Follow the guidelines in Section 10.4.10 for reporting dilutions.

NOTE: It is possible to dilute the surrogates in the sample extract below the linear range of the calibration curve. The minimum extract aliquot that can be used to provide a quantifiable result for the surrogates and matrix spikes is 0.0025mL (2.5uL).

SOIL: 10g to 10mL MeOH	WASTES: 1g to 10mL MeOH	Surrogates- Theoretical ng on-column
125uL(0.125mL)	100uL (0.100mL)	250
62.5uL(0.0625mL)	50uL(0.050mL)	125
25uL(0.025mL)	25uL(0.020mL)	50
12.5uL(0.0125mL)	10ul(0.010mL)	25
2.5uL(0.0025mL)	2.0uL(0.0020mL)	5.0-quantiation limit
<2.5uL(0.025mL)	<2.0uL(0.0020mL)	<5.0ng- below the quantitation limit-diluted out

NOTE: Some instrument quantitation limits may be higher than the limit listed in the table. The volume of extract should be adjusted accordingly.

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis of Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1 Two criteria must be met in order to identify a target compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{retention\ time\ of\ the\ target\ compound}{retention\ time\ of\ the\ associated\ internal\ standard}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. These ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.2 Tentatively Identified Compounds

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification. Tentative identifications of non-targets will be made only by analysts having completed the training specified in the training schedule.

11.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.2.2 The relative intensities of the major ions should agree within +/-30%.

11.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.

11.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

11.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria is not met exactly, the peak may be considered tentatively identified. The analyst should consult other analysts or the mass spectral interpretation specialist if there are any questions concerning an interpretation of spectra.

11.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculation is

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes DF$$

where
Cis = concentration of the internal standard, ug/L
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
DF = dilution factor

Soils by Heated P/T

$$TIC\ (ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{5.0g}{(W)(solids)}$$

where
Cis = concentration of the internal standard, ug/kg
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
W = weight of sample analyzed, g
solids = decimal equivalent of percent solids

Soils by Methanol Extraction

$$TIC\ (ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{Vcal}{(W)(solids)}$$

where
Cis = concentration of the internal standard, ug/kg
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = decimal equivalent of the percent solids(percent solids/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

where
Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)

11.3 Calculations for Samples-Internal Standard Technique

Aqueous Samples- relative response factor :

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes DF$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/L)
RRFavg = average response factor of the compound being measured
DF = dilution factor

Aqueous Samples: regression curve

$$concentration(ug/L) = concentration(curve) \otimes DF$$

where
DF = dilution factor

The reporting limit (RL) is calculated:

$$RL(ug/L) = RLqap \otimes DF$$

where
DF = dilution factor. The SL CQAP Table 5 RL(RLqap) assumes a DF of 1.

Soils by Heated P/T- relative response factor :

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{5.0g}{(W)(solids)}$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/kg)
RRFavg = average response factor of the compound being measured
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

Soils by Heated P/T: regression curve

$$conc(ug/kg,dw) = Ccurve(ug/kg) \otimes \frac{5.0g}{(W)(solids)}$$

where
Ccurve = concentration from curve(ug/kg)
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The reporting limit (RL) is calculated:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The STL-SL CQAP assumes W= 5.0g and solids = 1.

Methanol Extraction Soils and Wastes- relative response factor

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{Vcal}{(W)(solids)}$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/L)
RRFavg = average response factor of the compound being measured
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = (percent solids)/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)

Methanol Extraction of Soils and Solids- regression curve:

$$conc(ug,kg,dw) = Ccurve(ug/L) \otimes \frac{Vcal}{(W)(solids)}$$

where
Vcal = volume that calibration curve is based on (0.005L or 0.025L)
W = weight of sample added to the reagent water (g)-defined above

The reporting limit (RL)is calculated:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the reagent water (g)
solids = (percent solids)/100)

The STL-SL CQAP assumes W= 5.0g and solids = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in Section 3.1.3 of STL-SL SOP AN02: *Analytical Batching*. Note that the method blank for liquid samples and low-level soils is clock-specific and that the method blank for medium level soil samples is extraction batch-specific.

STL-SLSOP AN02: *Analytical Batching* describes the procedure for evaluating batch-specific QC. This criteria is summarized in the attached 8260 SOP Summary.

STL-SL SOP AN02 also contains the calculations for accuracy and precision and the calculations for the theoretical concentrations of surrogates, lab spikes, and matrix spikes.

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must demonstrate competence in the analysis of samples by this procedure. The minimum criteria for this demonstration is the preparation and analysis of spiked reagent water. Section 8.3 of EPA Method 8260A gives the general procedure for the performance of the IDOC and Table 6 of EPA Method 8260A gives the acceptance criteria for the accuracy and precision.

12.3 Method Detection Limit

The method detection limit is determined in accordance with STL-SL SOP CA90.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date. Section 10 of the STL-SL QAPs provide guidance on preventive maintenance.

14.0 TROUBLE-SHOOTING

Trouble-shooting items will be added at a later time. See instrument manufacturers' manuals for guidance on locating and repairing instrument problems.

15.0 REFERENCES

1. *Savannah Laboratories' Comprehensive Quality Assurance Plan* and *Savannah Laboratories' Corporate Quality Assurance Plan*, current revisions.
2. Method s 5035, 8000B, and 8260B. *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846.including Update III* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

Appendix A

VOLATILES BY GC/MS WORKING STANDARDS -EXAMPLE

These standards can be used to prepare the working standards for EPA Method 8260 to report the TCL (target compound list) compounds and the extended list of target compounds generally associated with EPA 8260. The standards are prepared in purge and trap grade methanol and are stored at 4C with minimum headspace.

Working Standard 1 (TCL WS-1)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA Cal #2	2000	12.5	25
VOA Cal #3	2000	12.5	25
VOA Cal #4	2000	12.5	25
1,2,-DCB	5000	5.0	25
1,3-DCB	5000	5.0	25
1,4-DCB	5000	5.0	25
2-CEVE	1000	125	125

Working Standard 2 (TCL WS-2)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA Cal #1	5000	25	125
8260 Surrogates	2500	10	25

Working Standard for GASES (TCL GASES)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
502.2 Cal 1	2000	12.5	25

Appendix A

Working Standard 3 (8260 WS-3)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
8260 Custom Mix #1	200	125	25
8260 Custom Mix #2	200	125	25
1,1,2,2-Tetrachloroethane	2000	12.5	25

Appendix A

Internal Standard (8260 ISTD)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
VOA ISTD	2500	20	50
1,2-DCE-d4	2000	25	50

Internal Standard/Surrogate (8260 ISSU)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA ISTD	2500	20	50
1,2-DCE-d4	2000	25	50
8260 Surrogate	2500	20	50

Tune Evaluation Standard (4-BFB)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
4-BFB	5000	10	50

Matrix Spike Standard (5-component subset)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
Matrix Spiking Solution	2500	20	50

TCLP matrix Spike Standard (5-component subset)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
TCLP Spiking Solution	2000	16	125

Appendix A

VOLATILES BY GC/MS CALIBRATION STANDARDS - EXAMPLES

The following calibration standards are prepared to define the working range of the EPA 8260 analysis for the target compound list (TCL) and the extended list of compounds generally associated with EPA 8260. The lowest level standard is at the reporting limit and the other standards define the working range. Samples with target analytes above the concentration of the highest calibration standard must be diluted and reanalyzed.

TARGET COMPOUND LIST

Working Level standards	Conc (ug/mL)	TCL-1 *	TCL-2 *	TCL-3 *	TCL-4 *	TCL-5 *	TCL-6 *
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 5.0mL of reagent water or to 5.0g of blank sand.

8260 EXTENDED LIST (TCL+ADDITIONAL COMPOUNDS)

Working Level standards	Conc (ug/mL)	8260-1 *	8260-2 *	8260-3 *	8260-4 *	8260-5 *	8260-6 *
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
8260 WS-3	25	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 5.0mL of reagent water or to 5.0g of blank sand.

CONCENTRATIONS OF THE CALIBRATION STANDARDS-5.0mL OR 5.0g

Cal Std	all targets except ketones, 2-CEVE	ketones, 2-CEVE
TCL-1,8260-1	5ug/l-kg	25ug/l-kg
TCL-2,8260-2	10ug/l-kg	50ug/l-kg
TCL-3,8260-3	25ug/l-kg	125ug/l-kg
TCL-4,8260-4	50ug/l-kg	250ug/l-kg
TCL-5,8260-5	100ug/l-kg	500ug/l-kg
TCL-6,8260-6	200ug/l-kg	1000ug/l-kg

Appendix A

VOLATILES BY GC/MS CALIBRATION STANDARDS-25mL Purge Volume-EXAMPLES

These calibration standards are prepared to define the working range of the EPA 8260 analysis for the target compound list (TCL) and the extended list of compounds generally associated with EPA 8260. The standards are based on a volume of 25mL to achieve lower quantitation limits for the target compounds. The lowest level standard is at the reporting limit and the other standards define the working range. Samples with target analytes above the concentration of the highest calibration standard must be diluted and reanalyzed.

TARGET COMPOUND LIST

Working Level standards	Conc (ug/mL)	25TCL -1*	25TCL-2*	25TCL-3*	25TCL-4*	25TCL-5*	25TCL-6*
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 25mL of reagent water.

8260 EXTENDED LIST (TCL+ADDITIONAL COMPOUNDS)

Working Level standards	Conc (ug/mL)	258260-1*	258260 -2*	258260-3*	258260-4*	258260-5*	258260-6*
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
8260 WS-3	25	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 25mL of reagent water.

CONCENTRATIONS OF THE CALIBRATION STANDARDS

Cal Std	all targets except ketones, 2-CEVE	ketones, 2-CEVE
25TCL-1,25-8260-1	1.0ug/l	5.0ug/l
25TCL-2,25-8260-2	2.0ug/l	10ug/l
25TCL-3,25-8260-3	5.0ug/l	25ug/l
25TCL-4,25-8260-4	10ug/l	50ug/l
25TCL-5,25-8260-5	20ug/l	100ug/l
25TCL-6,25-8260-6	40ug/l	200ug/l

Appendix B
8260 SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage*	Container	Hold Time
Aqueous	None; 4C	40mL no headspace	7 days
	HCl pH<2; 4C	40mL-no headspace	14 days
Soil/solid(low level)	Iced at collection; 5mL sodium bisulfate added upon arrival in lab; store at 4C	5-g Encore Sampler	14 days
Soil/solid(low level) -high carbonates	Iced at collection; 5mL water added upon arrival in lab; store at –10C	5-g Encore Sampler	14 days
Soil/solid(high level)	None; 4C	Glass 125mL	14 days
TCLP	HCl pH<2; 4C	Tedlar bag or syringe	14 days

*storage temperature is 4C with a control criteria of less than 6C with no frozen samples

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
4-BFB 50ng on column Clock starts at injection	4-BFB 50ng on column Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification (50ug/L or 50ug/kg)) RL Standard-low point on cal curve (if necessary)
Method blank	Method blank
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

See SL SOP AN02, Section 3.1.3, for the batch/clock options for LCS and MS/MSD.

Recommended Internal Standards:
1,2-dichloroethane-d4; 1,4-difluorobenzene; chlorobenzene-d5; 1,4-dichlorobenzene-d4

Surrogates/System Monitoring Compounds:
dibromofluoromethane; toluene-d8; 4-bromofluorobenzene

LCS/MS: CQAP Subset:
1,1-dichloroethene; benzene; trichloroethene; toluene; chlorobenzene

Appendix B
8260 SOP SUMMARY

VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION BROMOFLUOROBENZENE (BFB)	
m/e	**Abundance Criteria**
50	8.0-40.0% of mass 95
75	30.0-66.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	50-120%% of mass 95
175	4.0-9.0% of mass 174
176	93.0-101.0% of mass 174
177	5.0-9.0% of mass 176

(1) *8260 criteria taken from CLP OLMO4.0 (January 1998)

CALIBRATION ACCEPTANCE CRITERIA

Calibration Check Compounds - CCC
Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	Continuing Calibration
Less than or equal to 30% RSD	Less than or equal to 20% difference or drift from initial calibration

System Performance Check Compounds-SPCC

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30 (0.10 for 25mL purge volume)

See Sections 10.3 and 10.4 for ICAL and CCV linearity checks and criteria.

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
MS Tune Check - 50ng 4-BFB	Before initial and continuing calibration standards - every 12 hours	Mass abundances within method acceptance criteria	-Evaluate chromatogram and spectrum - Reanalyze - Retune MS and reanalyze - Remake standard and reanalyze - Perform instrument maintenance and reanalyze
Initial Calibration – minimum five point curve with lowest point at or below the Reporting Limit (RL)	Initially; after major instrument maintenance; whenever continuing calibration check fails. Prior to analysis of method blank and samples	Method criteria for CCC/SPCC (see -Calibration Acceptance Criteria – Table presented earlier in this document)	- Evaluate chromatograms, spectra, and integrations - Reanalyze standard(s) - Remake and reanalyze standard(s) - Perform instrument maintenance and recalibrate
Continuing Calibration check - midpoint standard	Every 12 hours before analysis of method blank and samples	Method criteria for CCC/SPCC (see Calibration Acceptance Criteria - Table presented earlier in this document)	- Evaluate chromatogram, spectra, integrations - Reanalyze standard - Remake and reanalyze standard - Recalibrate - Perform instrument maintenance and recalibrate
Method Blank	Every 12 hours (per clock) before sample analyses	All reported targets <RL	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
Lab Control Sample (LCS) -subset of target compounds unless full target spike specified by client	Each batch	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Matrix Spike/Matrix Spike Duplicate (MS/MSD) -subset of target compounds unless full target spike specified by client	Each batch	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Surrogates	All samples, blanks, LCS, MS	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Internal Standard Area	Evaluate all standards and samples	-Areas in continuing calibration verification must be 50% to +200% of previous initial calibration sequence -Retention time of internal standard must be +/-30 seconds from internal standard in initial calibration -Areas in samples should be evaluated for gross error . Consult supervisor.	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
Reporting Limit Standard -1x to 2x the RL	(Optional) Daily. Required for Florida DEP	Detected with reasonable response	-Evaluate chromatogram, spectra, and integrations -Reanalyze -Remake standard and reanalyze -Retune and recalibrate -Perform instrument maintenance and recalibrate
Initial Demonstration of Capability	Per analyst	Method criteria	-Reanalyze targets that do not meet criteria
Method Detection Limit (MDL)	See STL-SL SOP CA90	See STL-SL SOP CA90	-Reanalyze and re-evaluate

SEMI-VOLATILE COMPOUNDS BY GC/MS
Method: 8270C

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Approved by:

R. Wayne Robbins — 30 Aug 2002
Date

Title: Technical Manager

STL X Savannah __ Tallahassee __ Mobile __ Tampa West

1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine the concentration of various semi-volatile organic compounds (SVOC) in groundwater, TCLP and SPLP leachates, soils, sediments, wastes, and solid sample extracts. The attached quantitation report (Appendix B) lists the routine target compounds, the retention times of the target compounds, the characteristic ions of the target compounds, and the internal standard associated with each target compound.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits for the target compounds are given in Section 5 of the current revision of the Laboratory Quality Manual (LQM).

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using an appropriate extraction procedure. The extract is dried, concentrated to a volume of 1.0mL, and analyzed by GC/MS. Qualitative identification of the target compounds in the extract is based on the retention time and the mass spectra determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.2 This procedure is based on the guidance provided in SW-846 Method 8270C.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The toxicity or carcinogenicity of each chemical used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and lab glasses or face shield should be worn while handling extracts and standards. Standard preparation, addition of the internal standard solution, and sample extract dilution should be performed in a hood or well ventilated area.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst. These sheets specify the type of hazard that each chemical poses and the procedures that are used to handle these materials safely.

3.4 The exit vent of the splitless injector must have a carbon trap in-line to collect the semivolatile compounds that are vented during the injection of the extract. The traps should be changed every six months and disposed of in accordance with SOP CA70: *Waste Management.*

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult. Elevated baselines may be due to oils, greases, or other hydrocarbons that may be extracted from improperly cleaned glassware or extraction vessels.

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. Sample extracts that contain high concentrations of non-volatile material such as lipids and high molecular weight resins and polymers may require the optional GPC cleanup prior to analysis. The GPC cleanup is generally not effective in removing non-target material that is associated with common petroleum products like diesel.

4.3 Secondary ions may be used for quantification if there is interference with the primary quantitation ion. If a secondary ion is used for quantification, the concentration/response relationship of the secondary ion must be established. The secondary ion must meet the same calibration criteria as the primary ion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days from TCLP leaching procedure	40 days

Refrigerator temperature acceptance criterion is less than 6C with no frozen samples.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph- Hewlett-Packard (HP) 5890 or equivalent with compatible autosampler, splitless injector, and direct capillary interface. The exit vent of the splitless injector must have a carbon trap in-line to collect the semivolatile compounds that are vented during the injection of extracts. The carbon traps should be changed every six months.

6.2 Mass spectrometer- HP5971, HP5972, HP5973 or equivalent

6.3 Recommended Capillary column-HP-5MS, 30m x 0.25mm ID x 0.25um film thickness or equivalent column

6.4 Data system- compatible with GC/MS system

6.5 Microsyringes- appropriate volumes

6.6 Volumetric flasks- Class A, appropriate volumes

6.7 Autosampler vials and crimper- compatible with autosampler

7.0 REAGENTS

Reagents must be tracked in accordance with SOP AN44: *Reagent Traceability.*

7.1 Methylene chloride- pesticide residue grade, for preparation of standards

7.2 Acetone- pesticide residue grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SOP AN41: *Standard Material Traceability*. General guidance on the preparation of standards is given in SOP AN43: *Standard Preparation.*

The lab should purchase certified solutions from STL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SOP AN43 for guidance for standard preparation from neat materials.

8.1 Preparation of the Stocks from Neat Standards

The steps for the preparation of primary stock standards from neat materials are given in SOP *AN43: Standard Preparation.* The standards should be prepared in methylene chloride but may require other solvents to dissolve the material.

8.2 Preparation the calibration standards from the stock standards

A minimum of five calibration standards are prepared. The concentrations of the stock standards are in the 1000-10000ug/mL range. The recommended standards are listed in Section 10.2. The lowest level standard should be at the equivalent of the reporting limit and the rest of the standards should define the working range of the detector. Note that six calibration levels are required for a second order regression curve. Internal standards should be added to each standard to give a final concentration of 40ug/mL.

Each lab should develop controlled recipes that can be posted or maintained in appropriate logbooks.

9.0 SAMPLE PREPARATION

9.1 The sample extraction procedures are given in the following SOPs:

Matrix	SOP	Extraction Technique
Aqueous, TCLP leachates	EX30	Continuous Liquid-liquid Extraction
Aqueous, TCLP leachates	EX35	Separatory Funnel
Soils/Sediments	EX40	Sonication
Wastes	EX42	Waste dilution

9.2 The sample concentration procedures are given in SOP EX 50: Zymark Nitrogen Concentration.

9.3 Gel permeation chromatography (SOP EX61) may help to eliminate or minimize matrix interferences in a limited number of samples. The GPC cleanup is generally not effective on samples containing petroleum products.

10.0 PROCEDURE

10.1 Instrument Conditions

Instrument conditions may vary according to the sensitivity of each instrument. The following conditions are provided for guidance. The lab must optimize and document the conditions used for the analysis of SVOC by GC/MS.

Recommended Column:

HP-5MS 30m x 0.25mm ID x 0.25um film thickness or equivalent

Column flow: Approximately 1mL/min helium

GC Oven temperatures:

Initial column temperature: 45 C for 3 minutes
Column temperature program: 10C per minute
Final column temperature: 300C (until at least one minute past the elution time of Benzo (g,h,i) perylene).

GC injector parameters

Injector temperature: 250-270EC
Injection type: split, approximately 1:10 or splitless injection
Injector liner: 4mm ID quartz or 4mm glass, deactivated (single "Gooseneck")
Sample injection volume: 1-2uL

Mass Spectrometer and interface parameters

Mass spectrometer interface: 300C
Mass spectrometer source temperature: Factory Set
Mass range: 35-500amu, with a scan time of 1.0 scans per second or greater

10.2 Calibration

A minimum of five calibration standards are prepared and analyzed. The recommended standards are 10, 20, 50, 80, 100, 200ug/mL. The lowest level standard should be at or below the equivalent of the reporting limit and the rest of the standards should define the working range of the detector. Note that six calibration levels are required for a second order regression curve.

10.2.1 Fifty nanograms of DFTPP must be analyzed at the beginning of each 12-hour clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria demonstrates that the instrument is measuring the proper masses in the proper ratios. The DFTPP analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of DFTPP. All other instrument conditions must be identical-the mass range, scan rate, and multiplier voltage.

10.2.1.1 Prepare a 50ng/uL solution of tune/column evaluation standard containing each of the following compounds at 50ug/mL in methylene chloride: DFTPP, pentachlorophenol, p,p'-DDT, and benzidine.

10.2.1.2 Analyze a 1uL aliquot of the tune/column evaluation solution.

10.2.1.3Evaluate the DFTPP peak.

-The chromatogram should exhibit acceptable baseline behavior and the DFTPP peak should be symmetrical.

-The spectrum of the DFTPP must meet the criteria listed in the SOP Summary (Appendix A). Background subtraction must be straightforward, that is, no scan within the elution window of DFTPP may be subtracted from another scan within the elution window, and designed only to eliminate column bleed or instrumental background. Scans +/- 2 scans from the apex can be evaluated for the DFTPP criteria. Consecutive scans within this range may be averaged to meet the criteria.

NOTE: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

10.2.1.4Benzidine and pentachlorophenol should be present at their normal responses with minimal peak tailing visible. Peak tailing guidance is taken from EPA Method 625 which allows pentachlorophenol to be less than or equal to five and benzidine less than or equal to three. Refer to Figure 1for an example of [eak tailing factor calculation.

This is a good check on the system: if pentachlorophenol (a CCC) does not respond well, the calibration standard should not be analyzed. Injector port and column maintenance should be performed and the tune/column evaluation standard reanalyzed.

The percent breakdown of p,p'- DDT is calculated using the following equation. The percent breakdown should not exceed 20%.

$$\%Breakdown = \frac{(areaDDE + areaDDD)}{(areaDDT + areaDDE + areaDDD)} x\ 100$$

Areas from the total ion chromatogram are used to calculate DDT breakdown.

10.2.2 After the DFTPP criteria and column evaluation criteria have been met, the initial calibration standards are analyzed.

10.2.2.1Prepare the initial calibration standards. The lowest calibration standard should be at the RL and the rest of the standards will define the working range. See section 10.2 for guidance regarding calibration levels.

10.2.2.2Set up a sequence and analyze the calibration standards. The injection volume must be the same for the calibration standards and all sample extracts.

10.2.3 Identify the internal standards, surrogates, and the target compounds. The data system must be updated with the proper retention times and ion data.

10.2.4 Calculate the relative response factor for each compound as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured
(See the attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration of the compound being measured (ug/mL)
Cis = concentration of the internal standard (40ug/mL)

Secondary ions may be used for quantification if there is interference with the primary quantitation ion. If a secondary ion is used for quantification, the concentration/response relationship of the secondary ion must be established. The secondary ion must meet the same calibration criteria as the primary ion.

10.2.5 Calculate the average relative response factor (RRF_{avg}) for each target compound and each surrogate compound:

$$RRF_{avg} = \frac{RRF1 + RRF2 + RRF3.... + RRFn}{n}$$

RRF1 = relative response factor of the first standard
RRFn = relative response factor of the last standard
n = number of calibration standards

10.2.6 Calculate the standard deviation (SD) for the initial calibration standards:

$$SD = \sqrt{\frac{\sum_{i-1}^{n_1} (RRF_i - RRF_{avg})^2}{n-1}}$$

10.2.7 Calculate the relative standard deviation (%RSD) of the target compounds in the calibration standards.

$$\%RSD = \frac{SD}{RRF_{avg}} x\ 100$$

10.2.8 Evaluation of the Initial Calibration

The initial calibration is evaluated specifically for the calibration check compounds (CCC) and the system performance check compounds (SPCC). The CCC and SPCC criteria are given in the SOP Summary (Appendix A). The %RSD criteria for CCC and minimum RRF for SPCC must be met before the analysis of sample extracts can begin.

If the CCC and SPCC criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the initial calibration must be repeated. The analyst must be aware of the 12-hour clock for the DFTPP analysis. The DFTPP criteria must be met prior to the analysis of the calibration standards.

10.2.9 After the initial calibration criteria (CCC/SPCC) have been met, each target is evaluated for linearity. Refer to SOP AN67: *Evaluation of Calibration Curves* for guidance.

If the %RSD of the target compound is less than or equal to 15%, the average response factor can be used for quantitation of samples.

If the %RSD of the target compound is greater than 15%, a regression curve (linear, quadratic, etc) must be used for the quantitation of samples. A regression curve may also be used for the compounds that have %RSD less than 15%. The results can be used to plot a calibration curve of response ratios-Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where:

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See attached quantitation report for a list of the compounds and their associated internal standard)
Cx = concentration of the target compound being measured (ug/mL)
Cis = concentration of the internal standard (ug/mL)

A linear or quadratic curve may be used to define the concentration/response relationship. If r^2 is greater than 0.99, the curve can be used to quantify samples. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire concentration range.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

When more calibration levels are analyzed than required, individual compounds may be eliminated from the lowest or highest calibration levels(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of the calibration curve be eliminated without eliminating the entire level.

8000B exception: evaluation of the "grand mean": If the average %RSD of ALL (all targets including CCC and SPCC) compounds in the initial calibration is less than 15%, the average response factor can be used for quantitation of all target compounds. The recommended course is to use regression curves, as described above, to quantify targets where the %RSD criterion (<=15%) is exceeded.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

10.3 Continuing Calibration Verification

At the beginning of each 12-hour clock, the tune of the instrument must be checked by the analysis of the tune/column evaluation solution (10.2.1.1). The tune and column evaluation criteria (10.2.1.3 and 10.2.1.4) must be met before the analysis of the calibration check standards can take place.

10.3.1 After the tune and column evaluation criteria have been met, a continuing calibration check standard(s) is analyzed. The continuing calibration standard should be at a mid-level concentration. The CCC and SPCC criteria (SOP Summary, Appendix A) must be met before the analysis of samples can take place. The percent difference (%D) is calculated as follows:

$$\%D = \frac{RRFavg - RRFccv}{RRFavg} \otimes 100$$

where
RRFavg = average response factor from initial calibration
RRFccv = response factor from the check (12-hour) standard-calibration verification

The percent drift (%Drift) may also be used to evaluate the change/deviation of the curve:

$$\%Drift = \frac{Ci - Cccv}{Ci} \otimes 100$$

where
Ci = Calibration Check Compound standard concentration (ug/mL)
Cccv = measured concentration using the selected quantitation method (ug/mL)

NOTE: The SPCC criteria (10.3.8) must be met even if the regression curve option is used for quantitation. If these criteria are not met, corrective action must be taken. The corrective action may include reanalysis of the calibration check standard or preparation of a new secondary stock standard and reanalysis of the calibration check standard. If subsequent analysis of the standard is still out of criteria, a new initial calibration curve must be analyzed and evaluated.

10.3.2 The continuing calibration verification standard (CCV) must also be evaluated for internal standard response.

If the extracted ion current profile (EICP) area for any of the internal standards in the CCV changes by more than a factor of two (-50% to +100%) from the last initial calibration sequence, the analytical system must be inspected for problems and corrective action instituted.

10.4 Samples are analyzed only after the DFTPP criteria, column evaluation criteria and the calibration verification criteria have been met. The analytical system must be evaluated every 12 hours by the analysis and evaluation of the tune/column evaluation standard and a mid-level calibration standard.

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
Tune/Column Evaluation Standard Clock starts at injection	Tune/Column Evaluation Standard Clock starts at injection
Calibration standards- Minimum of five cal levels	Mid point calibration verification Optional RL: Standard-low point on cal curve
Samples analyzed until 12-hour clock expires	Samples analyzed until 12-hour clock expires

10.4.1 Remove the sample extracts to be analyzed from the refrigerator and allow the sample to come to ambient temperature.

10.4.2 Add 20-uL of the internal standard mix (2000 ug/mL) to each 1.0mL aliquot of the sample extract. The concentration of the internal standard in the extract is 40 g/mL.

10.4.3 Mix the contents of the autosampler vial by inverting several times.

10.4.4 Analyze the samples using the same analytical conditions used for the initial and continuing calibration standard. Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

NOTE: Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client. For TCLP analyses, every reasonable effort should be made to achieve the regulatory level with out instrument overload.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with t the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower RLs.

10.4.5 The dilution factor is calculated by dividing the volume of sample extract in microliters into 1000. For example, if 100uL of a sample extract are diluted to final volume of 1.0mL, the dilution factor is 10. (1000/100 = 10). The following table gives some dilution factors:

Dilution Preparation

uL extract-Vext	uL MeCl2	volume of dilution (Vdil-uL)	uL ISTD (2000ug/mL)-Vistd	DF
1000	0	1000	20	1
500	500	1000	10*	2
200	800	1000	16*	5
100	900	1000	18*	10
50	950	1000	19*	20
20	980	1000	20*	50

*assumes dilution of a 1mL extract or 1mL aliquot of an extract that has been spiked with the internal standard at 40ug/mL using 20ul of a 2000ug/mL internal standard solution

The concentration of internal standards must remain constant for all extracts and extract dilutions at 40ug/mL. The following equation can be used to determine the volume of the 2000ug/mL internal standard solution to add to an extract when a dilution is prepared from an extract that has already been spiked with the internal standard solution:

$$Vistd(uL) = 20uL - \left(\frac{Vext}{Vdil} \otimes 20ul\right)$$

Vistd = volume of 2000ug/mL internal standard to add to the diluted extract (uL)
Vext = volume of extract used to prepare the dilution (uL)
Vdil = final volume of the dilution (uL)-1000uL (1.0mL)

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis

11.1.1 Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from the daily calibration standard or a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1.1 Two criteria must be met in order to positively identify a compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{\textit{retention time of the target compound}}{\textit{retention time of the associated internal standard}}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. Ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.1.2 Tentatively Identified Compounds (TICs)

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification.

The default procedure is to evaluate up to 20 compounds of greatest apparent concentration that are not included as target compounds or routinely reported volatile compounds. The unknown compounds are tentatively identified using a forward search of the reference library.

If the library search produces a match at or above 85%, report that compound. If the library search produces more than one compound at or above 85%, report the first compound (the highest match quality). If the library search produces no matches at or above 85%, report the compound as unknown. If possible, provide a general classification of the unknown – for example, unknown aromatic, unknown hydrocarbon, etc.

TICs should be evaluated within the retention time range from the first eluting target or surrogate (whichever is first in the target list) to three minutes after the elution of the last target compound.

11.1.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.1.2.2 The relative intensities of the major ions should agree within +/-20%.

11.1.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.

11.1.2.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

11.1.2.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.1.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria is not met exactly, the peak may be considered tentatively identified. The analyst should consult senior analysts or the mass spectral interpretation specialist if there are any questions concerning an interpretation of spectra.

11.1.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculations assume that the same volume is injected for standards and samples.

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{F}{V} \otimes DF$$

where:

Cis =	concentration of the internal standard (ug/mL)
AREAis =	total ion peak area of the internal standard
AREAtic=	total ion peak area of the TIC
F =	final volume of extract (mL)
V =	volume of sample extract (L)
DF =	dilution factor

Soils

$$TIC\ (ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{F}{(W)(solids)} \otimes DF$$

where:

Cis =	concentration of the internal standard, ug/mL
AREAis =	total ion peak area of the internal standard
AREAtic=	total ion peak area of the TIC
F =	final volume of extract mL
W =	weight of sample analyzed (kg)
solids =	decimal equivalent of percent solids

11.2 Calculations for Samples-Internal Standard Technique
These calculations assume that the same volume is injected for standards and samples and that the standards and samples have the same concentration of internal standard.

11.2.1 Aqueous Samples

11.2.1.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{V} \otimes DF$$

where:

Ax =	area of the characteristic ion of the compound being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

11.2.1.2 If a regression curve is used, the concentration is given:

$$concentration(ug/L) = Ccurve \otimes \frac{F}{V} \otimes DF$$

where:

C_{curve} =	concentration from curve (ug/mL)
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

11.2.1.3 The reporting limit (RL) for each sample is given:

$$RL(ug/L) = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Vqap}{V} \otimes DF$$

where:

F = final volume of extract (mL)
Fqap = 1.0mL
Vqap = 1.0L
V = volume of sample extracted
DF = dilution factor. The LQM RL assumes a DF of 1.

NOTE: If V = 800mL to 1200mL, assume that Vqap/ V = 1 in the calculation of the reporting limit.

11.2.2 Soils

11.2.2.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ax =	area of the characteristic ion of the compound being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
W =	weight of sample extracted (kg)
solids =	(percent solids)/100
DF =	dilution factor

11.2.2.2 If the regression curve is used, the concentration is given:

$$conc(ug/kg,dw) = Ccurve \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ccurve =	concentration from curve(ug/mL)
W =	weight of sample extracted (kg)
F =	final volume of extract (mL)
solids =	(percent solids)/100)
DF =	dilution factor

11.2.2.3 The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Wqap}{(W)(solids)} \otimes DF$$

where

F = final volume of extract (mL)
W = weight of sample extracted (kg)
solids = (percent solids)/100

The LQM assumes Wqap = 30g, solids = 1, Fqap = 1.0mL, and DF = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in AN02: *Analytical Batching.* SOP AN02 also describes the procedure for evaluating batch-specific QC. The QA/QC criteria are summarized in the SOP Summary (Appendix A).

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must participate in the analysis of samples by this procedure in accordance with SOP CA92: *Evaluation of IDOCs.*

12.3 Method Detection Limit

The method detection limit is determined in accordance with SOP CA90: *Procedure for the Determination of the Method Detection Limit.*

13.0 PREVENTIVE MAINTENANCE & TROUBLESHOOTING

Refer to SOP AN53: *Preventive Maintenance Procedures for Laboratory Instruments* for guidance.

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Refer to SOP CA70: *Waste Management* for proper waste handling procedures.

15.0 REFERENCES

15.1 *STL Savannah Laboratory Quality Manual* current revision.

15.2 Method 8270C:*Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

APPENDIX A
8270C SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days	40 days

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
Tune/Column Evaluation Standard Clock starts at injection	Tune/Column Evaluation Standard Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification standard RL Standard (lowest point on calibration curve if required by client or state-specific QAP)
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION (DFTPP)	
m/e	**Ion Abundance Criteria (1)**
51	30-80% of mass 442
68	Less than 2.0% of mass 69
69	Present
70	Less than 2.0% of mass 69
127	25-75% of mass 198
197	Less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5.0-9.0% of mass 198
275	10-30% of mass 198
365	Greater than 0.75% of mass 198
441	Present but less than mass 443
442	40-110% of mass 198
443	15.0-24.0% of mass 442

(1) 8270 criteria taken from CLP OLMO4.0 (January 1998). The use of alternate criteria is expressly allowed in SW-846 Method 8270C.

APPENDIX A
8270C SOP SUMMARY

CALIBRATION ACCEPTANCE CRITERIA

Calibration Check Compounds - CCC
Phenol, 1,4-Dichlorobenzene, 2-Nitrophenol, 2,4-Dichlorophenol, Hexachlorobutadiene, 4-Chloro-3-methylphenol, 2,4,6-Trichlorophenol, Acenapthene, N-Nitrosodiphenylamine, Pentachlorophenol, Fluoranthene, Di-n-octylphthalate, Benzo(a) pyrene

System Performance Check Compounds-SPCC
N-Nitrosodi-n-propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol, 4-Nitrophenol

Initial Calibration	Continuing Calibration*
CCC: <= 30% RSD	CCC: <= 20% difference from initial calibration
SPCC: RRFavg >= 0.050	SPCC: RRF>= 0.050

*If CCC and/or SPCC do not meet the stated criteria, all targets that are reported must meet the CCC criteria.

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

The results for all target compounds are evaluated for linearity. If the %RSD is less than 15%, the calibration is assumed linear through the origin and the average response factor can be used for quantitation. If the average response factor for the target exceeds 15% (including any CCC), the analyst must use the calibration curve option.

NOTE: The lab has the option of using a regression curve for all analytes.

A linear, quadratic, or higher order regression fit may be used to define the concentration/response relationship. If r^2 is greater than 0.99, the curve can be used to quantify samples. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range. The minimum number of calibration standards required for a regression curve are given in the following table:

Type of curve	Minimum Number of Calibration Points
Linear (first order)	5
Quadratic (second order)	6

QC Item	Frequency	Acceptance Criteria	Corrective Action
Tune/Column Evaluation Standard DFTPP 50ng Pentachlorophenol - 50ng Benzidine – 50ng p,p'-DDT 50ng	Prior to analysis of calibration standards every 12 hours	DFTPP - within criteria	-Evaluate alternative scans -Reanalyze and evaluate -Retune and reanalyze -Clean source, retune, reanalyze
		Pentachlorophenol and benzidine - present at usual response with no peak tailing visible p,p'-DDT - %breakdown <20%	-Reanalyze -Perform injector port maintenance and reanalyze -Cut more than usual length of column and reanalyze -Replace column
Initial Calibration	After Tune Check and when calibration verification standard fails acceptance criteria. All initial calibration standards	CCC: %RSD < 30% SPCC: RRFavg > 0.050 Use regression curve for quantitation if %RSD for any target compound exceeds 15%	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Continuing Calibration Verification	After tune check; every 12 hours prior to analysis of samples	CCC: %Difference <= 20% Or %Drift <= 20% SPCC: RRF >= 0.050	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Internal Standard Areas	Evaluate all standards and samples	Areas in continuing calibration verification must be 50% to +200% of previous initial calibration sequence Areas in samples should be evaluated for gross error. Consult supervisor Retention time of internal standard must be +/-30 seconds from internal standard in previous CCV.	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available -Recalibrate

QC Item	Frequency	Acceptance Criteria	Corrective Action
Surrogate recovery	Evaluate for all samples and QC items if extract is not diluted OR If diluted, where >RL	Within LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Method Blank	Per batch	All targets < RL in LQM	-Evaluate chromatogram, spectra, and Integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
Lab Control Standard (LCS) - QAP subset	Per batch See SOP AN02	Within LQM Control Limits	-Evaluate chromatogram, spectra, and Integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
Matrix spike (MS) Matrix spike duplicate (MSD)	Per batch if sufficient sample volume/weight supplied See SOP AN02	Within LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
RL Standard (reporting limit)	Daily (optional)-lowest point on calibration curve if required by client or state-specific QAP	Detected at reasonable sensitivity	-Evaluate integrations and spectra; -Reanalyze -Prepare new standard and reanalyze
Initial Demonstration of Capability (IDOC)	Each work group	Accuracy and precision within method specified criteria	-Evaluate data -Reanalyze extracts if warranted -Re-extract and reanalyze for targets that fail criteria
Method Detection Limit (MDL)	Annually for each routine matrix See SOP CA90	Evaluate according to SOP CA90	Evaluate according to SOP CA90

APPENDIX B- TARGET COMPOUNDS

ROUTINE TARGET LIST

PARAMETER	RT	Quant Ion	Secondary Ions		ISTD
1,4-Dioxane	1.894	88	58	45	1
Pyridine	2.123	79	52	51	1
N-Nitrosodimethylamine	2.102	42	74		1
Aniline	3.812	93	66		1
Phenol	3.796	94	66	65	1
Bis(2-chloroethyl)ether	3.854	63	93	95	1
2-Chlorophenol	3.908	128	130	64	1
1,3-Dichlorobenzene	4.025	146	148	111	1
1,4-Dichlorobenzene	4.073	146	148	111	1
Benzyl Alcohol	4.202	108	79	77	1
1,2-Dichlorobenzene	4.239	146	148		1
2-Methylphenol	4.314	107	108	77	1
bis(2-Chloroisopropyl)ether	4.335	45	121		1
N-Nitroso-di-n-propylamine	4.469	70	42	101	1
3&4-Methylphenol	4.447	107	108		1
Hexachloroethane	4.522	117	201	199	1
Nitrobenzene	4.602	77	123	65	2
Isophorone	4.837	82	95	138	2
2-Nitrophenol	4.923	139	109	65	2
2,4-Dimethylphenol	4.965	107	122	121	2
Bis(2-chloroethoxy)methane	5.067	93	123	95	2
Benzoic acid	5.115	105	122		2
2,4-Dichlorophenol	5.169	162	164	98	2
1,2,4-Trichlorobenzene	5.259	180	182	145	2
Naphthalene	5.323	128	129		2
4-Chloroaniline	5.409	127	129	65	2
Hexachlorobutadiene	5.532	225	223	227	2
4-Chloro-3-methylphenol	5.991	107	144	142	2
2-Methylnaphthalene	6.135	142	141		2
1-Methylnaphthalene	6.269	142	141		2
Hexachlorocyclopentadiene	6.429	237	235	272	3
2,4,6-Trichlorophenol	6.541	196	198	200	3
2,4,5-Trichlorophenol	6.590	196	198	200	3
2-Chloronaphthalene	6.760	162	164	127	3
2-Nitroaniline	6.958	65	92	138	3
Dimethylphthalate	7.268	163	194	164	3
2,6-Dinitrotoluene	7.353	165	89	63	3
Acenaphthylene	7.337	152	151	153	3
3-Nitroaniline	7.540	138	108	92	3
Acenaphthene	7.599	154	153	152	3
2,4-Dinitrophenol	7.685	184	63	154	3
4-Nitrophenol	7.824	65	109	139	3
Dibenzofuran	7.829	168	139		3
2,4-Dinitrotoluene	7.914	165	89	63	3
2,3,4,5-Tetrachlorophenol	8.064	232	230	131	3
2,3,4,6-Tetrachlorophenol	8.091	232	230	131	3
Diethylphthalate	8.310	149	177	150	3

Fluorene	8.336	166	165	167	3
4-Chlorophenyl-phenylether	8.363	204	141	206	3
4-Nitroaniline	8.454	138	108	92	3
4,6-Dinitro-2-methylphenol	8.513	198	105	121	4
N-Nitrosodiphenylamine	8.555	169	168	167	4
1,2-Diphenylhydrazine	8.593	77	105	182	4
4-Bromophenyl-phenylether	9.090	248	250	141	4
Hexachlorobenzene	9.293	284	142	249	4
Pentachlorophenol	9.581	266	264	268	4
Phenanthrene	9.784	178	176	179	4
Anthracene	9.854	178	176	179	4
Carbazole	10.137	167			4
Di-n-Butylphthalate	10.847	149	150	104	4
Fluoranthene	11.659	202	203	101	4
Benzidine	11.926	184	92	185	5
Pyrene	12.006	202	200	203	5
Butylbenzylphthalate	13.214	149	91	206	5
3,3'-Dichlorobenzidine	13.892	252	254	126	5
Benzo(a)Anthracene	13.866	228	229	226	5
Bis(2-ethylhexyl)phthalate	14.111	149	167	279	5
Chrysene	13.924	228	226	229	5
Di-n-octylphthalate	14.971	149	43		5
Benzo(b)fluoranthene	15.367	252	253	125	6
Benzo(k)fluoranthene	15.399	252	253	125	6
Benzo(a)pyrene	15.783	252	125	253	6
Indeno(1,2,3-cd)pyrene	17.284	276	139		6
Dibenzo(a,h)anthracene	17.317	278	139	279	6
Benzo(g,h,i)perylene	17.674	276	277	138	6
SURROGATES					
2-Fluorophenol	3.032	112	64		1
Phenol-d5	3.785	99	71		1
Nitrobenzene-d5	4.586	82	128	54	2
2-Fluorobiphenyl	6.643	172	171		3
2,4,6-Tribromophenol	8.732	330	332	141	3
Terphenyl-d14	12.332	244	122	212	5
INTERNAL STANDARDS					
1,4-Dichlorobenzene-d4	4.057	152	150	115	1
Naphthalene-d8	5.302	136	68		2
Acenaphthene-d10	7.556	164	162	160	3
Phenanthrene-d10	9.747	188	94	80	4
Chrysene-d12	13.887	240	236	120	5
Perylene-d12	15.858	264	265	260	6

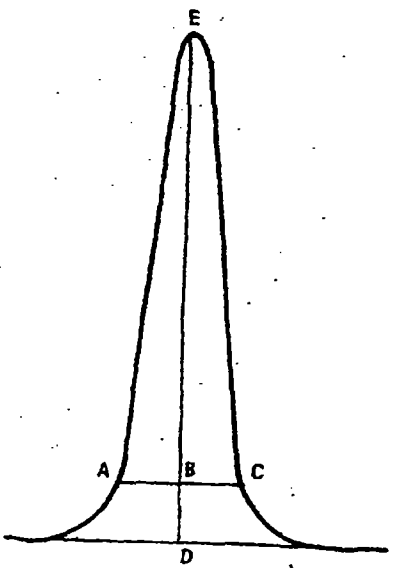
APPENDIX B- TARGET COMPOUNDS

APPENDIX IX TARGET LIST

PARAMETER	RT	Quant Ion	Secondary Ions			ISTD
1,4-Dioxane	1.933	88	58	45		1
Pyridine	2.178	79	52	51		1
2-Picoline	2.664	93	66			1
1,4-Benzoquinone	4.466	54	108	82		1
N-Nitrosomethylethylamine	2.739	88	42	43	56	1
Methyl methanesulfonate	2.964	80	79	65		1
N-Nitrosodiethylamine	3.268	102	42	44	57	1
Ethyl methanesulfonate	3.503	79	109	97	45	1
N-Nitrosopyrrolidine	4.481	100	41	42		2
Acetophenone	4.486	105	77	51		2
N-Nitrosomorpholine	4.497	56	86			1
O-Toluidine	4.529	106	107	79		2
Phorate	9.123	75	121			4
N-Nitrosopiperidine	4.796	114	42	55	56	2
O,O,O-Triethyphosphorothioate	5.122	65	97	93		2
2,6-Dichlorophenol	5.469	162	164	98		2
Hexachloropropene	5.507	213	211	215	117	2
1,2,4,5-Tetrachlorobenzene	6.447	216	214	179		3
,a-Dimethylphenethylamine	5.619	58	91	42		2
N-Nitroso-di-n-butylamine	5.875	84	57	41		2
1,4-Phenylenediamine	5.864	108	80	107		2
Safrole	6.094	162	104	135	103	2
Isosafrole	6.757	162	104	131		2
1,1-Biphenyl	6.805	154	76			3
1,4-Naphthoquinone	7.056	158	104	76		3
m-Dinitrobenzene	7.317	168	76	50		3
Pentachlorobenzene	7.916	250	248	252	215	3
1-Naphthylamine	8.001	143	115	116		3
2-Naphthylamine	8.113	143	115	116		3
2,3,4,6-Tetrachlorophenol	8.140	232	230			3
5-Nitro-o-toluidine	8.466	152	77	106		3
Thionazin	8.471	107	96	97		4
Sulfotepp	9.032	97	65			4
1,3,5-Trinitrobenzene	9.091	213	74	120		4
1-Diallate	9.118	86	43	234		4
Phenacetin	9.161	108	109	179		3
2-Diallate	9.235	86	43	234		4
Dimethoate	9.396	87	93	125		4
4-Aminobiphenyl	9.556	169	168	170		4
Pronamide	9.748	173	175	145		4
Pentachloronitrobenzene	9.748	237	295	142	214	4
Disulfoton	9.935	88	60			4
Dinoseb	9.957	211	163	147		4
thyl parathion	10.517	109	125			4
Nitroquinoline-1-oxide	11.094	174	101	128	75	4
Parathion	11.158	109	97			4
Famphur	13.178	218	93	125		4

Methapyrilene	11.388	97	58	191		4
Aramite-1	12.435	185	191	319		5
Aramite-2	12.563	185	191	319		5
p-Dimethylaminoazobenzene	12.638	120	225	77		5
Chlorbenzilate	12.745	139	251	75		4
Kepone	17.739	272	270	237		5
3,3'-Dimethylbenzidine	13.167	212	196	106		5
2-Acetylaminofluorene	13.562	181	180	223		5
7,12-Dimethylbenz(a)anthracene	15.438	256	239	241		6
Hexachlorophene	15.747	196	198			6
3-Methylcholanthrene	16.324	268	252	253		6
INTERNAL STANDARDS						
1,4-Dichlorobenzene-d4	4.102	152	150	115		1
Naphthalene-d8	5.346	136	68			2
Acenaphthene-d10	7.601	164	162	160		3
Phenanthrene-d10	9.802	188	94	189		4
Chrysene-d12	13.926	240	236	120		5
Perylene-d12	15.902	264	265	260		6

FIGURE 1 – Tailing Factor Calculation



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

POLYCHLORINATED BIPHENYLS (PCBs) by GC/MS

(Method: EPA 680)

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Technical Approval:

Andrea Teal 05/28/04
Date

Title: QA Manager
STL Savannah

Safety Approval:

Ernest B Weston 05/27/04
Date

Title: EHSC
STL Savannah

1.0 SCOPE AND APPLICATION

1.1 This procedure is used to determine the concentration of polychlorinated biphenyls (PCBs) in groundwater, soils, sediments, wastes, and biological tissues by GC/MS. PCBs are reported by the level of chlorination: monochlorobiphenyls, dichlorobiphenyls, trichlorobiphenyls, etc., up to decachlorobiphenyl.

1.2 The routine target analytes, reporting limits (RL), method detection limits (MDL), and the accuracy and precision limits are given in the current revision of the *Laboratory Quality Manual* (LQM) prepared by and for STL Savannah.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 Summary of Method

A measured volume or weight of sample is spiked with a surrogate and extracted using an appropriate extraction procedure. The extract is dried, concentrated to a volume of 1.0mL, and analyzed by GC/MS operated in the Selected Ion Monitoring Mode (SIM). Windows are established to monitor for the characteristic masses of the various PCB homologues. Qualitative identification of the target compounds in the extract is based on the presence of the peak within the SIM window and the mass ratio between the primary and confirmation ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion. Results are reported as total monochlorobiphenyls, total dichlorobiphenyls, etc.

The default identification and quantitation procedure will be to use only the quantitation and confirmation ions for the PCB homologues. Interference check ions, as described in Section 11.2.3, will not be used routinely to evaluate peaks as PCB homologues unless specified in a client QAP or agency requirement or the sample concentration is near a critical quantitation limit. Samples evaluated according to the default quantitation procedures may be slightly high biased.

2.2 Definitions - Refer to SOP AN99: *Definitions, Terms, and Acronyms* for a complete listing of applicable definitions.

Congener: a member of a family or class of compounds such as polychlorinated biphenyls (PCBs). There are 209 congeners of PCBs
Homologue: a PCB isomer with the same level of chlorination; e.g., the monochlorobiphenyl isomers, the dichlorobiphenyl isomers, etc.

2.3 This procedure is based on the guidance provided in EPA Method 680.

2.4 Method Modifications

The method has been modified to include the use of a carbon-13 labeled analogue of decachlorobiphenyl (13C12-DCB) as the surrogate in place of the labeled BHC and DDT compounds. 13C12-DCB can be subjected to the optional acid cleanup (the unlabeled analogue, decachlorobiphenyl, is routinely used in SW-846 Method 8082). A window-defining mix containing the first and last eluting isomers of each level of chlorination is used as an aid to establish and verify that the SIM windows are properly set.

This SOP contains preparation and sample evaluation procedures for soils and biological tissues, which are not included in EPA Method 680. The CCV criteria for soils and biota have been broadened to 30%D due potential matrix interferences associated with these types of samples, which may affect end-capping standards.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Waste Disposal SOP, and this document.

3.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Hexane is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult. Elevated baselines may be due to oils, greases, or other hydrocarbons that may be extracted from improperly cleaned glassware or extraction vessels

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. Sample extracts that contain high concentrations of non-volatile material such as lipids and high molecular weight resins and polymers may require the optional GPC cleanup prior to analysis. The GPC cleanup is generally not effective in removing non-target material that is associated with common petroleum products such as diesel or waste oil. GPC cleanup may be necessary for biological tissues. Acid cleanup may be employed as an additional cleanup tool.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

The following table lists the routine sample containers, storage conditions, and holding time associated with this procedure:

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time (1)	Extract Hold Time
Aqueous	none; 4°C	1-L amber	7 days	40 days
Soil/Sediment	none; 4°C	500-mL	14 days	40 days
Biological	Frozen	Glass or aluminum foil	6 months	40 days

(1) Holding times are advisory - no holding times are defined in method 680.

6.0 APPARATUS AND MATERIALS

6.1 GC/MS System with compatible data system, autosampler, splitless injector, and direct capillary interface.

6.2 Recommended Capillary column: HP-5MS, 30m x 0.25mm ID, 0.25um film thickness. Equivalent columns can be used.

6.3 Microsyringes: appropriate volumes

6.4 Volumetric flasks, Class A: appropriate volumes

6.5 Analytical balance

7.0 REAGENTS

The preparation of reagents must be performed in accordance with SOP AN44: *Reagent Traceability*.

7.1 Hexane – reagent grade

7.2 Acetone – reagent grade

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SOP AN41: *Standard Material Traceability*. General guidance on the preparation of standards is given in SOP AN43: *Standard Preparation*.

The lab should purchase certified solutions from STL-approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SOP AN43 for guidance for standard preparation from neat materials.

8.1 The recommended calibration standards are listed in Appendix A, Table 1. Prepare these standards at the stated concentrations in hexane.

8.2 The surrogate compound, 13C12-Decachlorobiphenyl, is prepared at a concentration of 2.5ug/mL. 1.0mL of this solution is spiked into all samples and QC items prior to extraction.

8.3 The matrix spiking solution contains one PCB congener of each chlorination level except for the nonachlorobiphenyls. The solution is prepared at the indicated concentrations in acetone. 1.0mL of this solution is spiked into all lab spikes and matrix spikes.

COMPOUND	CONCENTRATION (ug/mL)
2-Chlorobiphenyl	2.0
2,3-Dichlorobiphenyl	2.0
2,4,5-Trichlorobiphenyl	2.0
2,2',4,6-Tetrachlorobiphenyl	4.0
2,2',3,4,5'-Pentachlorobiphenyl	4.0
2,2',4,4',5,6'-Hexachlorobiphenyl	4.0
2,2',3,4',5,6,6'-Heptachlorobiphenyl	6.0
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	6.0
Decachlorobiphenyl	10

9.0 SAMPLE PREPARATION

9.1 The sample extraction procedures are given in the following SOPs:

Matrix	SOP Number	Extraction Technique
Aqueous	EX30	Continuous Liquid-liquid Extraction
Soils/Sediments and Biological	EX40	Sonication

9.2 The sample concentration procedures are given in SOP EX 50: *Zymark Nitrogen Concentration.*

9.3 Gel permeation chromatography may help to eliminate or minimize matrix interferences in a limited number of samples. The GPC cleanup is generally not effective on samples containing petroleum products. Acid cleanup (SOP EX60: *Acid, Permanganate, and Copper Cleanups for PCBs and Pesticides*) is recommended as a routine cleanup prior to analysis. Sulfur cleanup may be necessary if the sample extract contains high levels of sulfur.

10.0 ANALYTICAL PROCEDURE

10.1 Instrument Conditions
Instrument conditions may vary according to the sensitivity of each instrument. The following conditions are provided for guidance. The lab must document the conditions used for the analysis of SVOC by GC/MS.

Recommended Column:
HP-5MS 30m x 0.25mm ID, 0.25um film thickness or equivalent

Column flow:
Approximately 1mL/min helium

GC Oven temperatures:

Initial column temperature: 45°C for 1 minute
Column temperature program 1: 20°C per minute to 150°C, hold 1 minute
Column temperature program 2: 10°C per minute to 310°C, hold until DCB and 13C12-DCB elute

GC injector parameters:

Injector temperature: 250-260°C
Injector: splitless
Inlet purge time: 0.8 minutes
Injector liner: 4mm ID quartz or 4mm glass, deactivated
Sample injection volume: 1µL

Mass Spectrometer and interface parameters:

Mass spectrometer interface: 300°C
Mass spectrometer source temperature: Factory Set
Mass range: SIM (see Table 3 in Appendix B for ions to monitor)
Mass range for DFTPP analysis: 35-500amu at 1 scan per second or less.

10.2 Tune Criteria

Ten nanograms of DFTPP are analyzed at the beginning of each 12-hour clock as a check on the "tune" of the mass spectrometer. The DFTPP analysis is performed using scan analysis. The same tune parameters are used for the SIM analysis of the calibration standards and samples.

10.2.1 Prepare a 10ng/µL solution of DFTPP column evaluation standard. The standard must also contain p,p'-DDT (4,4'-DDT).

10.2.2 Analyze a 1µL aliquot of the 10ng/µL DFTPP solution using the same temperature program that is used for SIM analysis of the calibration standards, samples, and QC samples.

10.2.3 Evaluate the DFTPP peak.

-The chromatogram should exhibit acceptable baseline behavior and the DFTPP peak should be symmetrical.

-The spectrum of the DFTPP must meet the criteria listed in the SOP Summary. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans ± 2 scans from the apex can be evaluated for the DFTPP criteria. Consecutive scans within this range may be averaged to meet the criteria.

NOTE: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

10.3 Window-Defining Solution and SIM Parameters

10.3.1 Analyze 1µL of the window-defining solutions in the scan mode from 45amu to 500amu at > 1 scan per second. Use the same temperature program that will be used for the SIM analysis of PCBs. The window defining solutions may be analyzed separately or may be combined into a single solution.

10.3.2 Determine the retention times of the first and last eluting congeners at each level of chlorination. The quantiation and confirmation masses are listed in Table 3 of Appendix B.

10.3.3 Set the SIM parameters as follows. Refer to Table 3 of Appendix B for the ion sets.

-Begin data acquisition with ion set #1 before the elution of PCB congener #1, the first eluting CL-1-PCB
-Stop the acquisition of ion set #1 and begin acquisition of ion set #2 approximately 10 seconds before the elution of PCB congener #104, the first eluting CL5-PCB.
-Stop the acquisition of ion set #2 and begin the acquisition of ion set #3 approximately 10 seconds after the elution of PCB congener #77, the last eluting CL4-PCB.
-Stop the acquisition of ion set #3 and begin the acquisition of ion set #4 approximately 10 seconds after the elution of 4,4'-DDT. (The retention time of 4,4'-DDT is determined from the scan analysis of the DFTPP solution that is analyzed at the beginning of each 12-hour clock.)
-Stop the acquisition of ion set #4 and begin the acquisition of ion set #5 approximately 10 seconds before the elution of PCB congener #208, the first eluting CL9-PCB.

10.4 Initial Calibration
Initial calibrations are performed in accordance with SOP AN67: *Evaluation of Calibration Curves.* After the SIM windows are established and verified and the DFTPP criteria have been met, the initial calibration standards are analyzed. Note that a single PCB congener of each chlorination level is used for calibration and quantitation. Decachlorobiphenyl is used to quantify nonachlorobiphenyls.

10.4.1 Prepare the initial calibration standards. The lowest calibration standard should be at the RL, and the rest of the standards will define the working range.

10.4.2 Set up a sequence and analyze the calibration standards. The injection volume must be the same for the calibration standards and all sample extracts. The routine volume is 1µL.

~~10.4.3 Identify the internal standards, surrogates, and the target compounds. The data system must be updated with the proper retention times and ion data.~~

10.4.4 The relative response factor for each compound is calculated as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where

Ax = area of the characteristic ion of the calibration congener
Ais = area of the characteristic ion for Chrysene-d10
Cx = concentration of the compound being measured (µg/mL)
Cis = concentration of the internal standard (40µg/mL)

NOTE: Use Chrysene-d10 as the internal standard unless matrix interferences are encountered. If phenthrene-d10 must be used, the calibration must be re-evaluated and verified using the second internal standard.

10.4.5 Calculate the average relative response factor (RRF_{avg}) for each target compound and each surrogate compound:

$$RRF_{avg} = \frac{RRF1 + RRF2 + RRF3 \ldots + RRFn}{n}$$

RRF1 = relative response factor of the first standard
RRFn = relative response factor of the last standard
n = number of calibration standards

10.4.6 Calculate the standard deviation (SD) for the initial calibration standards:

$$SD = \sqrt{\frac{\sum_{i-1}^{n_1} (RRF_i - RRF_{avg})^2}{n-1}}$$

10.4.7 Calculate the relative standard deviation (%RSD) of the target compounds in the calibration standards:

$$\%RSD = \frac{SD}{RRF_{avg}} x\,100$$

- If the %RSD of each target compound is less than or equal to 20%, the average response factor can be used for quantitation of samples.
- If the %RSD of the target compound is greater than 20%, the curve should be evaluated for errors and one or more standards re-analyzed. Take corrective action until the %RSD of each target is less than 20%.

10.4.8 Performance Criteria
In addition to meeting the calibration criteria, the following performance criteria must also be met for the mid-level standard.

-Mass abundance ratios of all calibration congeners within acceptance range
(See Table 4 Appendix B)
-Baseline separation of PCB congener #87 from congeners #154 and #77, which may coelute.
-Signal-to-noise ratio of >=5 for decachlorobiphenyl ion 499 and chrysene-d12 ion 241
-decachlorobiphenyl mass abundance: mass 500 >=70% but <=95% of mass 498

10.5 Continuing Calibration Verification
Samples are analyzed only after the DFTPP criteria and the calibration acceptance criteria have been met. The analytical system must be evaluated every 12 hours by the analysis and evaluation of the DFTPP and a mid-level calibration standard prior to the analysis of samples and after the samples by the analysis and evaluation of a mid-level standard. The endcap standard must be analyzed within the 12 hour clock.

10.5.1 The percent difference or percent drift between the continuing calibration RRF and the average relative response factor (RRFavg) is calculated for each target compound and each surrogate compound:

$$\%difference = \left| \frac{RRF - RRFavg}{RRFavg} \right| \otimes 100$$

where
RRF = relative response factor from CCV
RRFavg = average relative response factor from initial calibration curve

If the percent difference is less than or equal to 20% for each target compound, the initial calibration is verified. If soils and/or biota are being evaluated, the CCV criteria have been broadened to 30%D due potential matrix interferences associated with these types of samples, which may affect end-capping standards.

If the continuing calibration criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the analysis of the continuing calibration standard must be repeated. The analyst must be aware of the 12-hour clock DFTPP criteria must be met prior to the analysis of the calibration standards. If the continuing calibration standard repeatedly fails the calibration verification criteria, the initial calibration curve must be reanalyzed and reevaluated.

The performance criteria given in Section 10.4.8 must also be met prior to the analysis of samples.

10.6 Sample Analysis
Remove the sample extracts to be analyzed from the refrigerator and allow the sample to come to ambient temperature.

10.6.1 Add 30µL of the internal standard mix (25µg/mL) to each 1-mL aliquot of the sample extract. The concentration of the internal standard in the extract is 0.75ng/µL.

10.6.2 Mix the contents of the autosampler vial by inverting several times.

10.6.3 Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

10.6.4 The dilution factor is calculated by dividing the volume of sample extract in microliters into 1000. For example, if 100µL of a sample extract is diluted to final volume of 1.0mL, the dilution factor is 10 (1000/100 = 10). The following table gives some dilution factors:

Dilution Preparation

µL extract-Vext	µL MeCl2	volume of dilution (Vdil-µL)	µL ISTD (25µg/mL)-Vistd	DF
1000	0	1000	30	1
500	500	1000	15*	2
200	800	1000	24	5
100	900	1000	27	10
50	950	1000	28.5	20
20	980	1000	30	50

*assumes dilution of a 1.0mL extract or 1mL aliquot of an extract that has been spiked with the internal standard at 0.75µg/mL using 30ul of a 25.0µg/mL internal standard solution

The concentration of internal standards must remain constant for all extracts and extract dilutions at 0.75µg/mL. The following equation can be used to determine the volume of the 25.0µg/mL internal standard solution to add to an extract when a dilution is prepared from an extract that has already been spiked with the internal standard solution:

$$Vistd(uL) = 30\mu L - \left(\frac{Vext}{Vdil} \otimes 30\mu l\right)$$

Vistd = volume of 25.0ug/mL internal standard to add to the diluted extract (µL)
Vext = volume of extract used to prepare the dilution (µL)
Vdil = final volume of the dilution (µL)-1000µL (1.0mL)

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Analysis

The default procedure will be to use only the quantitation and confirmation ions for identification and quantification of PCB congeners. Interference check ions, as described in Section 11.2.3, will not be used routinely to evaluate peaks as PCB congeners unless specified in a client QAP or agency requirement or the sample concentration is near a critical quantitation limit.

11.1.1 Examine the Selected Ion Current Profiles (SICP) for the internal standards. Confirm that the RT and response of the internal standards are within the acceptance criteria specified in the SOP Summary. If the internal standard retention times have changed significantly or the peaks cannot be located, stop and analysis and correct the problem. Reanalyze any associated samples.

11.1.2 Evaluate the peaks for candidates to be identified as PCBs. A peak is tentatively identified as a PCB if:

-The peak falls within the retention time range bordered by the first and last eluting isomer of that chlorination level

-The ratio of the quantitation and confirmation ions are present and the area ratios fall within the acceptance criteria in Appendix B, Table 4. The scans must maximize within one scan of each other. Examine the data for the presence of a coeluting PCB of higher chlorination if both ions and the M-70 ions are present and the ratio does fall within the acceptance limits.

-The areas for the quantiation and confirmation ions must be greater than three times the background noise and must fall within the working range of the calibration curve (must not saturate the detector

-At least one ion in the M-70 cluster must be present

11.1.3 Evaluate each PCB candidate in the Cl-3 to Cl-7 range for the presence of coeluting PCBs containing one or two additional chlorines. An intense M+35 ion at the retention time may indicate a PCB with one additional chlorine and the presence of an intense M+70 would indicate a co-eluting PCB containing two additional chlorines. Use the information in Tables 5 and 6 of Appendix B to correct for the interfering ion(s).

For example, if a Cl-7-PCB and a Cl-5-PCB coelute, the Cl-7-PCB will contribute to the quantiation and confirmation ions for the Cl-5-PCB. Cl-7-PCB produces a cluster of three ions by the loss of two chlorines-ions 322,324, and 326. Two of these ions, 324 and 326, are also ions contained in the molecular ion cluster of Cl-5-PCB. To determine the ion 326 and 324 areas produced only by the Cl-5-PCB, calculate the contribution to each and subtract it from the measured areas. See Tables 5 and 6 in Appendix B for the percentage of the interference peak to subtract from the quantiation and confirmation ions. In this example, 164% of the area measured for ion 322 should be subtracted from the area measured for ion 324 and 108% of the area measured for ion 322 should be subtracted from ion 326.

NOTE: A coeluting PCB with one more chlorine will affect only the quantiation ion (Table 6). The interference from a coeluting PCB containing one more chlorine, due to the natural abundance of 13C12, is small and can usually be neglected except when measuring the area of a small amount of a PCB coeluting with a large amount of a another PCB containing one more chlorine.

11.2 Calculations for Samples-Internal Standard Technique
These calculations assume that the same volume is injected for standards and samples.

11.2.1 Aqueous Samples

$$concentration(\mu g/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{V} \otimes DF$$

where

Ax =	sum of areas of the characteristic ion of the PCB chlorination level being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (µg/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

The reporting limit (RL) for each sample is given:

$$RL(\mu g/L) = RL_{LQM} \otimes \frac{F}{F_{LQM}} \otimes \frac{V_{LQM}}{V} \otimes DF$$

where

F =	final volume of extract (mL)
F_{LQM} =	1.0mL
V_{LQM} =	1.0L
V =	volume of sample extracted
DF =	dilution factor. The LQM (RL_{LQM}) assumes a DF of 1.

NOTE: If V = 800mL to 1200mL, assume that Vqap/ V = 1 in the calculation of the reporting limit.

11.2.2 Soils

$$concentration(\mu g/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ax =	sum of areas of the characteristic ion of the PCB chlorination level being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (µg/mL)

RRFavg = average response factor of the compound being measured
F = final volume of extract (mL)
W = weight of sample extracted (kg)
solids = (percent solids)/100
DF = dilution factor

The reporting limit (RL) for each sample is given:

$$RL = RL_{LQM} \otimes \frac{F}{F_{LQM}} \otimes \frac{W_{LQM}}{(W)(solids)} \otimes DF$$

where

F = final volume of extract (mL)
W = weight of sample extracted (kg)
solids = (percent solids)/100

The LQM assumes W_{LQM} = 30g, solids = 1, F_{LQM} = 1.0mL, and DF = 1.

12.0 QUALITY CONTROL AND DATA ASSESSMENT

12.1 Analytical Batching

QC data must be evaluated against the precision and accuracy criteria set forth in the Laboratory Quality Manual and SOP AN02: *Analytical Batching and Evaluation of QC Data*. SOP AN02 also provides guidance for establishing and evaluating QC items to be included in an analytical batch.

The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in SOP AN02. The attached SOP summary provides guidance for evaluating sample data.

12.2 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP CA85: *Nonconformance and Corrective Action Procedures*. CA85 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures.

13.0 METHOD PERFORMANCE

The Reporting Limits (RL), the Method Detection Limits (MDL), and accuracy and precision limits associated with these methods are given in the current revision of the Laboratory Quality Manual prepared by and for STL Savannah.

13.1 Initial and Continuing Demonstration of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP *CA92: Procedure for Initial and Continuing Analyst Demonstration of Capability*.

13.2 Method Detection Limit

The method detection limit must be determined for each analyte in accordance with SOP CA90: *Procedures for the Determination of Method Detection Limit (MDL)*.

14.0 PREVENTIVE MAINTENANCE AND TROUBLESHOOTING

Refer to SOP AN53: *Maintenance Procedures for Laboratory Instrumentation* for routine preventive maintenance and the manufacturer's guides for trouble-shooting items.

15.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Follow the guidance for disposal in SOP CA70: *Waste Disposal.* Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Excess samples, reagents, and standards must be disposed in accordance with SOP CA70: *Waste Management.*

- Flammable waste (hexane from extracts, rinsings, and standards) – Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full.

- Excess samples. Dispose according to characterization on sample disposal sheets. If non-hazardous, dispose down drain/sewer. If hazardous, transfer to hazardous waste department for storage.

- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

- Excess oil samples – Transfer to waste department for storage/disposal.

16.0 REFERENCES

~~STL Savannah's *Laboratory Quality Manual (LQM),* current revision~~

Severn Trent Laboratories' *Quality Management Plan (QMP), current revision*

Method 680: Determination of Pesticides and PCBs in Water and Soils/Sediment by Gas Chromatography/Mass Spectrometry. November 1985. Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, USEPA, Cincinnati, OH

17.0 TABLES, DIAGRAMS, AND VALIDATION DATA

Appendix A, B, and C contain applicable tables including the SOP and QC Summary.

APPENDIX A

TABLE 1 CALIBRATION STANDARDS

CALIBRATION COMPONENTS	CAL 1	CAL 2	CAL3	CAL4	CAL5
Calibration Congener					
2-chlorobiphenyl (1)	0.10	0.50	1.0	2.0	5.0
2,3-dichlorobiphenyl(5)	0.10	0.50	1.0	2.0	5.0
2,4,5-trichlorobiphenyl(29)	0.10	0.50	1.0	2.0	5.0
2,2',4,6-tetrachlorobiphenyl(50)	0.20	1.0	2.0	4.0	10
2,2',3,4,5'-Pentachlorobiphenyl (87)	0.20	1.0	2.0	4.0	10
2,2',4,4',5,6'-hexachlorobiphenyl(154)	0.20	1.0	2.0	4.0	10
2,2',3,4',5,6,6'-heptachlorobiphenyl(188)	0.30	1.5	3.0	6.0	15
2,2',3,3',4,5,5',6,6'-octachlorobiphenyl(200)	0.30	1.5	3.0	6.0	15
Decachlorobiphenyl	0.50	2.5	5.0	10	25
Retention Time Congeners					
3,3',4,4-tetrachlorobiphenyl(77)	0.20	1.0	2.0	4.0	10
2,2',4,6,6'-Pentachlorobiphenyl (104)	0.20	1.0	2.0	4.0	10
2,2',3,3',4,5,5',6,7'-nonachlorobiphenyl(208)	0.40	2.0	4.0	8.0	20
Surrogate					
13C12-Decachlorobiphenyl	0.50	2.5	5.0	10	25
Internal Standards					
Phenathrene-d10	0.75	0.75	0.75	0.75	0.75
Chrysene-d12	0.75	0.75	0.75	0.75	0.75

TABLE 2 –First and Last Eluting Isomers

Congener	First Eluting Isomer	Last Eluting Isomer
Cl-1	2-chlorobiphenyl	4-Chlorobiphenyl
Cl-2	2,6-dichlorobiphenyl	4,4'-dichlorobiphenyl
Cl-3	2,2',6-trichlorobiphenyl	3,4,4'-trichlorobiphenyl
Cl-4	2,2',6,6'-tetrachlorobiphenyl	3,3',4,4'-tetrachlorobiphenyl
Cl-5	2,2',4,6,6'-pentachlorobiphenyl	3,3',4,4',5-pentachlorobiphenyl
Cl-6	2,2',4,4',6,6'-hexachlorobiphenyl	3,3',4,4',5,5'-hexachlorobiphenyl
Cl-7	2,2',3,4',5,6,6'-heptachlorobiphenyl	2,3,3',4,4',5,5'-heptachlorobiphenyl
Cl-8	2,2',3,3',5,5',6,6'-octachlorobiphenyl	2,3,3',4,4',5,5',6-octachlorobiphenyl
Cl-9	2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl

APPENDIX B
SIM IONS

Table 3-Ions for SIM Acquisition

ION Set 1 (a)	ION Set 2 (b)	ION Set 3 (c)	ION Set 4 (d)	ION Set5 (e)
152	186	248	240	356
153	188	249	241	358
186	220	254	288	360
187	222	256	290	390
188	254	288	322	392
189	255	290	324	394
190	256	322	326	424
220	258	323	356	425
221	288	324	357	426
222	289	326	358	428
224	290	328	360	430
255	292	357	362	432
256	294	358	391	462
258	323	360	392	464
290	324	362	394	466
292	326	392	396	496
294	328	394	398	498
	358	396	428	499
	360	398	430	500
	362		432	502

(a) Cl-1 to Cl-4 and Phenthrene-d10
(b) Cl-3 to-Cl-6
(c) Cl5 to Cl-7
(d) Cl-6 to Cl-8 and Chrysene-d12
(e) Cl-8 to Cl-10 and 13C12-DCB

TABLE 4-Approximate Retention Times for PCB Isomer Groups and Calibration Congeners

PCB Isomer Group	Approximate RRT Range	Calibration Congener	Approximate Calibration Congener RRT
Cl-1	0.30-0.35	2-chlorobiphenyl-(1)	0.30
Cl-2	0.38-0.50	2,3-dichlorobiphenyl(5)	0.43
Cl-3	0.46-0.64	2,4,5-trichlorobiphenyl(29)	0.54
Cl-4	0.55-0.82	2,2',4,6-tetrachlorobiphenyl(50)	0.56
Cl-5	0.64-0.92	2,2',3,4,5'-entachlorobiphenyl (87)	0.80
Cl-6	0.75-1.1	2,2',4,4',5,6'-hexachlorobiphenyl(154)	0.82
Cl-7	0.88-1.2	2,2',3,4',5,6,6'-heptachlorobiphenyl(188)	0.88
Cl-8	0.99-1.21	2,2',3,3',4,5,5',6,6'-octachlorobiphenyl(200)	1.03
Cl-9	0.16-1.28	Decachlorobiphenyl	1.3
Cl-10	1.3	Decachlorobiphenyl	1.3

RRT = retention time relative to Chrysene-d12

Table 4 Quantitation and Interference Check Ions

PCB Isomer Group	Quant ION	Confirmation ION	Expected Ratio(a)	Acceptable Ratio(a)	M-70 Confirmation ION	Interference Check ION M+70	Interference Check ION M+35
Cl-1	188	190	3.0	2.5-3.5	152	256	222
Cl-2	222	224	1.5	1.3-1.7	152	292	256
Cl-3	256	258	1.0	0.8-1.2	186	326	290
Cl-4	292	290	1.3	1.1-1.5	220	360	326
Cl-5	326	324	1.6	1.4-1.8	354	394	360
Cl-6	360	362	1.2	1.0-1.4	288	430	394
Cl-7	394	396	1.0	0.8-1.2	322	464	430
Cl-8	430	428	1.1	0.9-1.3	356	498	464
Cl-9	464	466	1.3	1.1-1.5	390		498
Cl-10	498	500	1.1	0.9-1.3	424		
Chrysene-d12	240	241	5.1	4.3-5.9			
Phenathrene-d10	188	189	6.6	6.0-7.2			
13C12-DCB (surrogate)	510	512					

(a) ratio of quantitation ion to confirmation ion

TABLE 5-Corrections for Interference of PCB Containing Two Additional Chlorines

PCB Isomer Group	Quant ION	Confirmation ION	Ion Measured to Determine Interference	Percent Ion area to be subtracted from QUNAT ION Area	Percent Ion area to be subtracted from CONFIRMATION ION Area
Cl-3	256	258	254	99	33
Cl-4	292	290	288	65	131
Cl-5	326	324	322	108	164
Cl-6	360	362	356	161	71
Cl-7	394	396	390	225	123

TABLE 6-Corrections for Interference of PCB Containing One Additional Chlorine

PCB Isomer Group	Quant ION	Ion Measured to Determine Interference	Percent Ion area to be subtracted from QUNAT ION Area
Cl-2	222	221	13.5
Cl-3	256	255	13.5
Cl-4	292	289	17.4
Cl-5	326	323	22.0
Cl-6	360	357	26.5
Cl-7	394	391	30.9
Cl-8	430	425	40.0

APPENDIX C
680 SOP SUMMARY

HOLD TIME & PRESERVATION SUMMARY

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time (1)	Extract Hold Time
Aqueous	none; 4°C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4°C	500-mL	14 days	40 days
Biological	Frozen	Glass or aluminum foil	6 months	40 days

[1] Holding times are advisory - no holding times are defined in method 680.

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
DFTPP 10ng on column Clock starts at injection	DFTPP 10ng on column Clock starts at injection
Calibration standards- minimum of five cal levels	*Mid point calibration verification*
Samples and the capping standard must be analyzed within 12 hours of the start of the clock	Samples and the capping standard must be analyzed within 12 hours of the start of the clock
Capping standard	Capping standard

DFTPP CRITERIA

m/z	Ion Abundance Criteria
127	40-60% of mass 198
197	<1.0% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass443
442	>40% of mass 198
443	17-23% of mass 442

CALIBRATION ACCEPTANCE CRITERIA

Initial Calibration	Continuing Calibration
RRFavg <= 20% RSD	Percent difference <= 20% difference from initial calibration for aqueous samples; <=30% for soils and biota

QC Item	Frequency	Acceptance Criteria	Corrective Action
Tune/Column Evaluation Standard DFTPP 20ng	Prior to analysis of calibration standards every 12 hours	DFTPP - within criteria	-Evaluate alternative scans -Reanalyze and evaluate -Retune and reanalyze -Clean source, retune, reanalyze
Initial Calibration-minimum of five calibration standards	After Tune Check and when calibration verification standard fails acceptance criteria.	%RSD <= 20%	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Performance Criteria	Evaluate mid level calibration standard each clock	-Mass abundance ratios of all calibration congeners within acceptance range (see Appendix B) -Baseline separation of PCB congener #87 from congeners #154 and #77 -Signal-to-noise ratio of >=5 for decachlorobiphenyl ion 499 and chrysene-d12 ion 241 -decachlorobiphenyl mass abundance: mass 500 >=70% but <=95% of mass 498	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Continuing Calibration Verification	After tune check; every 12 hours prior to analysis of samples and at the end of the analytical sequence	%Difference <= 20% for aqueous samples; <=30% for soils and biota	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate

QC Item	Frequency	Acceptance Criteria	Corrective Action
Internal Standard Areas	Evaluate all standards and samples	Areas in continuing calibration verification must be within 30% of the previous CCV or within 50% of the initial calibration Areas in samples should be evaluated for gross error . Consult superior	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate recovery	Evaluate for all samples and QC items if extract is not diluted OR If diluted, where >RL	Within LQM limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Method Blank	Per batch	All targets < RL in LQM	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in SOP AN02
Lab Control Standard (LCS) - LQM subset	See AN02	All spiked targets within the accuracy criteria in LQM	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in SOP AN02
Matrix spike (MS) Matrix spike duplicate (MSD)	Per batch if sufficient sample volume/weight supplied See AN02	All targets within the accuracy and precision criteria in LQM	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in SOP AN02
Demonstration of Capability (DOC)	Initially and then annually per analyst	Accuracy and precision within method specified criteria	-Evaluate data -Reanalyze extracts if warranted -Re-extract and reanalyze for targets that fail criteria
Method Detection Limit (MDL)	Annually	Evaluate according to SOP CA90	Evaluate according to SOP CA90

STEVENSON TRENT STL

SOP SUMMARY FORM

SOP#: SM07.05.28.04.2	SOP Description: POLYCHLORINATED BIPHENYLS (PCBs) by GC/MS

Revisions:

___Minor _X_ Significant ___Complete Re-write ___New SOP

Summary of Revision(s):

- Revised format to be consistent with current STL Savannah SOP format and NELAC requirements
- Revised safety information to be consistent with current STL format.
- Clarified method modification section to include CCV requirements for soils and biota, section 2.4
- Removed reference to separatory funnel extraction procedure. No longer performed, section 9.1
- Changed injection volume from 2uL to 1uL, section 10.1
- Clarified SIM parameters in section 10.3.3
- Added 30% CCV criteria for soils and biota, section 10.5.1
- Added requirement to analyze endcap within 12-hour clock, section 10.5
- Added quality control, method performance, and waste management information
- Revised cal level 3 concentration in Table 1

IDOCs Required:	___Yes	_X__ No	___ NA
MDLs Required:	___Yes	_X__ No	___ NA

SOP Implementation Date: 06.28.04

Target Training Completion Date: 06.28.04

Approved by: Andrea Seal

Title: QA Manager

Date: 05/28/04

Division Approval:

Title: Laboratory Director

Date: 5/28/04

CHLORINATED HERBICIDES

(Methods: EPA 515.1, 615, and SW-846 8151A)

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Approval and Signatures

Quality Assurance Manager:	Andrea Teal	Date: March 21, 2005
Laboratory Director:	Benjamin Gulizia	Date: March 21, 2005
EH&S Officer:	Ernest Walton	Date: March 21, 2005

SOP REVISION SUMMARY

Revision Type:

___Minor _X_ Significant ___Complete Re-write ___New SOP

Summary of Changes Since Previous Revision:

- Revised format to be consistent with current STL Savannah SOP format and NELAC requirements
- Revised safety information
- Updated the type of columns used in this procedure
- Added information on herbicide standards and correction factors to Section 8
- Revised the GC parameters in Section 10
- Removed the option to use internal standard calibration for initial calibration of the GC; not performed
- Removed information about the RL standard, no longer used
- Added quality control, method performance, preventative maintenance, and waste management information
- Revised Retention Times for target compounds listed in Appendix A
- Revised GC conditions in Appendix A
- Replaced calibration standard tables in Appendix B
- Updated the Laboratory Performance Solution Criteria in Appendix C, also included in Method Modification in Section 2

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures used to determine the concentration of chlorinated herbicides in various matrices. Appendix A contains an example of the retention time order for the herbicides, Appendix B provides examples of the calibration standards routinely analyzed, and Appendix C contains a summary of the method QC requirements for Methods 515.1, 615, and 8151A.

Method	Routine Matrices
515.1	Drinking water
615	Water and wastewater
8151	Water, groundwater, soils, solids, wastes, leachates

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits are listed in the Laboratory Quality Manual (LQM) prepared by and for STL Savannah.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 *Environmental samples are prepared using the procedures outlined in SOP EX45: Extraction of Chlorinated Herbicides.* The extracted methyl derivatives are analyzed by a GC equipped with dual capillary columns (different phases) connected to dual electron capture (EC) detectors, allowing simultaneous detection and confirmation of the target compounds. Quantitation may be performed using the external or internal standard calibration technique.

2.2 GC/MS confirmation can also be employed if analyte concentration is sufficiently high or if the sample extract is concentrated to an appropriate final volume. The esterified extract must be used for the GC/MS confirmation – do not use the 8270 extract.

2.3 Method Clarifications/Default Procedures

Elimination of Calibration Points: When more calibration standards are analyzed than required, individual compounds may be eliminated from the lowest or highest concentration level(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of a calibration be eliminated without eliminating the entire level.

Bracketing Sample Extracts: The default procedure for continuing calibration verification for the 8000-series methods is to bracket samples by CCV standards (before and after) if external standard calibration is used and ***not*** to cap the sequence (run CCV after the samples) if internal standard calibration is used ***unless*** noted in the client QAPP or in an STL pre-project plan. The internal standard provides verification information on the sensitivity and retention time stability of the instrument and verification of acceptable injections of the sample extracts. See Appendix C for summaries of the analytical sequences.

Grand Mean: The "grand mean" is used to evaluate calibration data according to the provisions of SW-846 Method 8000B and Sections 10.3 and 10.4 of this SOP.

Dilutions: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve. For example, a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client. Project managers and lab staff must work together to balance client satisfaction with productivity.

2.4 This method is based on the guidance in SW-846 Methods 8000B, 8151A, 40 CFR 136 Method 615, and EPA method 515.1.

2.4 Method Modification:

EPA Method 515.1 requires the analysis of a Laboratory Performance Check (LPC). In addition to the sensitivity check for dinoseb, the EPA 515.1 LPC includes checks for chromatographic performance (using 4-Nitrophenol) and column performance (using 3,5-Dichlorobenzoic acid and 4-Nitrophenol). The laboratory does not report 4-Nitrophenol or 3,5-Dichlorobenzoic acid as these analytes are not regulated drinking water analytes; therefore, only the dinoseb sensitivity check is required by this SOP.

2.5 Definitions – Refer to SOP AN99: *Definitions, Terms, and Acronyms* for a complete listing of applicable definitions.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.1 Specific Safety Concerns or Requirements

Hexane is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

4.1 Glassware should be scrupulously cleaned and solvent-rinsed in accordance with SOP AN60: *Glassware Cleaning Procedures* to minimize artifacts and/or elevated baselines in gas chromatograms. Any vessel that comes in contact with the extract is a potential source for contamination. Method blanks that are extracted and analyzed with each batch of samples will provide clues to the source of contamination from the glassware and reagents.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The sample may require dilution prior to analysis to reduce or eliminate interferences. The extraction procedure SOP EX45 has several steps that are designed to eliminate or minimize interferences due to matrix. The extract is diluted as needed for data analysis. If a cleanup is used, the method blank and lab control standard must also be subjected to the cleanup.

4.3 Injection port maintenance is very important for the consistent detection of the reactive herbicides such as dinoseb.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Refer to Appendix C for a summary of the sample collection, storage, and preservation requirements.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph (GC), temperature programmable, equipped with dual electron capture (EC) detectors and a compatible autosampler

6.2 Data system compatible with the GC, with appropriate software or integration capabilities

6.3 The following column pairs are recommended. Other columns/phases may be used if the calibration and QC criteria are met and adequate separation of the target compounds is achieved.

J&W DB-XLB 30 M x 0.32 mm ID x 0.5 um film
J&W DB-35MS 30 M x 0.32 mm ID x 0.5 um film

6.4 Microsyringes: appropriate volumes

6.5 Volumetric flasks: Class A, appropriate volumes

6.6 Autosampler vials, septa, and caps: compatible with the autosampler

7.0 REAGENTS

The preparation of reagents must be performed in accordance with SOP AN44: *Reagent Traceability.*

Hexane - pesticide grade or equivalent, for preparation of standards

8.0 STANDARDS

8.1 The preparation of the calibration standards must be tracked in accordance with SOP AN41: *Standard Material Traceability.* General guidance on the preparation of standards is given in SOP AN43: *Standard Preparation.*

8.2 The lab should purchase certified solutions from STL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SOP AN43 for guidance for standard preparation.

8.3 Herbicide standards are purchased as methyl esters; therefore, the concentration of the standard must be corrected to the free acid concentration. This will eliminate the need to correct the final concentration of the sample. The correction factors are given in Appendix D.

8.4 Calibration Standard Recipes

The recipes used for standard preparation must be clearly documented as a controlled posting or as a narrative in the traceability log. The lowest level calibration standard should be at or below the equivalent of the reporting limit as defined in the LQM or client QAPP. The remaining standards will define the working range of the analytical system. Appendix B contains example recipes of the calibration levels for the routinely determined herbicides.

9.0 SAMPLE PREPARATION

The sample preparation and cleanup procedures are described in SOP EX45

10.0 ANALYTICAL PROCEDURE

10.1 Gas Chromatograph Operating Conditions

The instrument conditions listed in this section are for guidance. The actual conditions used by the lab must be documented in the instrument maintenance log, data system, or run log. The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

10.1.1 Two configurations are routinely used for the analysis of herbicides. A single column may be connected to the injection port or two columns may be connected to the injection port using a press-tight glass y-splitter and a guard column, a two-hole ferrule, or a glass tee to provide

simultaneous detection and confirmation of the target analytes.

10.1.2 Example GC Parameters

Columns:
J&W DB-XLB 30 M x 0.32 mm ID x 0.5 um film
J&W DB-35MS 30 M x 0.32 mm ID x 0.5 um film
Injector:240°C
Detector: 300°C
Carrier Gas Flow: Helium at ~2mL/min (per column) (pressure at 20psi, constant)
Make-up Gas Flow: Nitrogen at ~60mL/min (per detector)

Temperature program:

Initial Temp:	50°C
Initial Hold:	0.50 min
Program Rate 1	12°C/min to 100°C
HoldTemp 1:	15°C/min to 200°C
Program Rate 2	80°C/min
FinalTemp :	300°C (hold for 1.25 minutes)
TOTAL TIME	15.73 minutes

NOTE: These conditions and parameters are given for guidance. The columns/phases, GC conditions, and instrument parameters may be modified to optimize the analytical system.

10.2 Initial Calibration

Internal or external standard calibration techniques may be employed for the determination of the concentration of herbicides. Pentachloronitrobenzene (PCNB) may be a suitable compound to use as an internal standard

10.2.1 Prepare and analyze the calibration standards. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards. Guidance for establishing the analytical sequence is given in the SOP Summary.

Note that the following offers two options for calibration and quantitation – average CF or regression curve. Only one needs be chosen per analyte.

10.2.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the hexane blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed? Is the column properly seated in the injector and detector ports?
>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?
>Are the peaks symmetrical? Is there tailing or fronting?
>Are the areas of the peaks normal for the sensitivity setting being used?

Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

10.2.3 Evaluate the calibration curve in accordance with SOP AN67: *Evaluation of Calibration Curves.*

10.3 Initial Calibration Criteria:

Method 515.1: If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

Method 615: If the relative standard deviation is less than 10% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

Method 8151: If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

The preferred method of quantitation is the average response or calibration factor. If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve (Section 10.2.5). The "grand mean exception" described below should be applied to 8151A initial calibrations only in extraordinary circumstances because of the difficulty of maintaining and providing documentation on an on-going basis.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average calibration factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20% and no single compound has a %RSD greater than 60%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required since the lab has demonstrated that the lowest standard in the calibration curve (the equivalent of the RL) can be detected.

Regression Curve Option: A calibration curve is established for each analyte by plotting the concentration along the x-axis and the corresponding response along the y-axis. If r^2 is greater than 0.99, the curve can be used to quantify samples. For 8000-series methods, a minimum of five points is required for a linear regression, six points for a second order curve, and seven or more for higher order fits. It is recommended to use only linear and quadratic (second order) curves for quantitation. See SOP AN67 for guidance on evaluation of calibration curves.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

10.4 Calibration Verification

Calibration is verified at the frequency given in the SOP Summary. If external standard calibration is used, the following criteria apply to calibration standards analyzed before and after samples. In situations where compounds fail criteria high and no positive hits for the compound(s) failing high are detected, these samples may be reported.

If internal standard calibration is used, the samples do not have to be bracketed (capped) by the analysis of a CCV standard unless specified by a regulatory agency or client QAPP.

10.4.1 Analyze a mid-level standard. The concentration of the verification standard should be varied periodically to evaluate the calibration curve in the lower and upper halves. Tabulate the area of the target analytes and calculate the response factors if using the average RF/CF option. If using the calibration curve option, calculation of the RF is unnecessary.

Calculate the percent drift or percent difference between the initial and continuing calibration in accordance with SOP AN67.

10.4.2 Continuing Calibration Criteria

Response Criteria

If the CCV criterion is not met, another CCV should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the calibration verification criteria cannot be met, a new initial calibration must be prepared, analyzed, and evaluated.

Method 515.1: If the percent drift or percent difference is less than or equal to 20%, the initial calibration is verified and the average response factor or regression curve can be used for quantitation.

Method 615: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor or regression curve can be used for quantitation.

Method 8151: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor or regression curve can be used for quantitation.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average calibration factor or regression curve from the initial calibration can be used for quantitation if the average %drift or average % difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15% and no single compound is greater than 45%D.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

All samples analyzed using external standard calibration must be bracketed by acceptable CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

Retention Time Criteria
The retention time for the CCV must fall within the daily retention time window as defined in SOP AN66: *Determination of Retention Time Windows for Gas Chromatographic Analyses.*

Internal Standard Response Criteria
If internal standard calibration is used, the response of the internal standard(s) must be within -50% to +150% of the response in the CCV-level standard in the initial calibration sequence. If

the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

10.5 Sample Analysis Sequence

The analytical sequences for the methods are given in the SOP Summary in Appendix C. The default is to exclude QC items (method blanks, LCS, and MS/MSD) in determining the maximum number of extracts in the clock. For 8151A, more than 20 extracts (samples and QC) may be analyzed in a sequence, as long as the 12 hour time frame has not elapsed, but the number of samples (non-QC extracts) may not exceed 20. Note that some client and agency QAPPs may require that the QC items be counted as part of the twenty samples.

10.5.1 The sample extract is injected using the same injection volume used for the calibration standards. Extracts that are known to be relatively clean should be analyzed first. Extracts suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.5.2 If the concentration of target compounds exceeds the working range (defined by the highest standard in the initial calibration), the extract must be diluted in hexane and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve. If the internal standard calibration is used, the concentration of the internal standard in the diluted extract must be the same as in the calibration standards.

NOTE: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve. For example, a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client. Project managers and lab staff must work together to balance client satisfaction with productivity.

10.5.3 Occasionally, situations may arise where part of the chromatogram is obscured by large non-target peaks or matrix interferences (short, wide, peaks that are not well resolved). In these situations, it is permitted to report a lower RL for the target compounds that are not affected by the non-target or matrix interference and perform a dilution only for the target compounds that are affected. This anomalous situation must be discussed with the project manager and section supervisor prior to reporting the results and noted in the case narrative or anomaly report. Again, project managers and lab staff must work together to balance client satisfaction with productivity.

10.6 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SOP AN66: *Determination of Retention Time Windows and Evaluation of Retention Time Data Chromatographic Analyses.*

11.0 DATA ANALYSIS AND CALCULATIONS

Methyl ester herbicide standards must be corrected to the free acid concentration. This is performed by comparing the molecular weight of the methyl ester that of the acid ton determine a correction factor. Appendix D gives the molecular weights of the acids and esters. It also lists the correction factors and illustrates how to perform the acid-ester correction.

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times); the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration; and the integration of the peaks. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data. SOP AN66: *Determination of Retention Time Windows and Evaluation of Retention Time Data for Chromatographic Analyses* provides additional guidance for the evaluation of chromatographic data. This guidance is summarized in the following sections.

11.1 Compounds of Concern

Dalapon - this compound elutes very early in the run and may be subject to interference from co-eluting compounds and from artifacts from the extraction process.

MCPA and MCPP - these compounds have very low response in comparison to the other herbicides.

Dinoseb - this compound can be lost in the extraction process (hydrolysis step) but also may be lost if the injection port is not frequently and properly maintained.

11.2 Manual integrations must be documented in accordance with SOP AN65: *Manual Integrations*. Data systems should be adjusted to minimize operator intervention. All chromatographic peaks must be evaluated for overall peak shape and "reasonableness" of integration. Under no circumstances should manual integrations be used to change reasonable data system integrations in order to meet calibration or QC criteria.

11.3 The judgement and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks properly integrated?
- Are co-eluting peaks or matrix interferences present?
- Is the internal standard present at the correct retention time and response (-50% to150% of the response in the associated CCV)? Are the surrogates present at the expected RT or have they shifted?

Qualitative analysis

Identification of the surrogates and target compounds is based on retention time. The retention time (RT) windows calculated around the CCV retention times are used for the identification of the target compounds. The analyst should also note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target

compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect". This may only be performed when the RT of the internal standard and surrogates are at their respective retention times and there is little or no evidence of matrix interferences. If there is doubt as to whether the peak can be excluded or not, the default procedure will be to report the peak as the target compound unless another technique (for example, GC/MS) is used to determine that the target compound is not present.

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peak areas that exceed the established linear range of the calibration curve may result in significant retention time shifts; therefore, all peaks, which have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are handled using dilutions as detailed above (Section 10.5.2).

Evaluate the internal standard (if used) and the surrogates to check for shifts in retention times and to evaluate the surrogate recovery. The recovery criteria for surrogates are given in the LQM.

Internal Standard Criteria

The internal standard must be within the retention time window defined by the associated CCV. The response of the internal standard(s) must be within a range +/–50% of the response of the internal standard in the associated CCV.

If sample matrix interferences preclude the use of internal calibration for a sample extract, two options should be considered:

1) dilute the extract to minimize or eliminate the interference
2) use external standard calibration to quantify the target and surrogate compounds (if external standard calibration is used, all calibration requirements, including a capping standard, must be met - see Appendix C for the external standard sequence).

Surrogate Criteria

DCAA is used as the surrogate for herbicide analysis. Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Evaluate the surrogates in the same manner as the target compounds using the guidance in the table in Section 11.1.3.

NOTE: If the recovery of the surrogate(s) is above the upper control limit and no target compounds are detected in the sample, results may be reported. Refer to SOP AN02 regarding this issue.

Evaluate each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences, and the overall shapes of the peaks.

If the concentration is below the lowest calibration standard or MDL (if the sample is being evaluated for "J" results), the reporting limit (RL) for that compound is calculated (Section 11.2). The RL is calculated for all target compounds that are not detected on the primary analytical column. Peaks over-range are handled using dilutions as detailed above (10.5.2).

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected or if the concentration is within the calibration range with the %RPD >40, the analysis at a dilution is not necessary.

If the result for a target is above the reporting limit (RL) on the primary column, evaluate the confirmation column. Use the retention time window calculated using the CCV as guidance for the identification of the target compounds. Note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect".

If the target compound is detected on the confirmation column, the concentration of the target compound is calculated and compared to the result from the primary column. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

If the relative percent difference is less than or equal to 40%, the presence of the target compound is confirmed and the higher concentration is reported.

NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A larger relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. Flag the result to note the disparity (P flag) between the results. Alternatively, dilute the extract to a level that removes the interference and report the RL from this dilution.

The default guidance in this table assumes the following:
1) the retention time and response of the internal standard(s) are within acceptance criteria with little or no shift in RT
2) surrogate recovery meets the acceptance criteria and peaks fall within the middle of it's retention time window with little or no shift in RT
3) the peak identified as the target falls in the middle of the retention time window for that compound

Default Guidance for Evaluation of Surrogates and Target Compounds in Samples, LCS, and MS

PEAK INFORMATION	COLUMN 1	COLUMN 2	%RPD	REPORT
No peak present	No peak		NA	<RL
		No peak	NA	If compound is a surrogate, re-extract. If sample is LCS, re-extract.
Peak present at RT	<E	<E	<=40%	Report highest
	<E	<E	>40%	Report result most appropriate for sample matrix. Use lowest result as default. Flag with "P"
Peak present at RT	>E	<E	<=40%	Dilute extract to get both results within the calibration curve.
	<E	>E		
	>E	<E	>40%	Report lowest result and flag with "P"
	<E	>E		No dilution required.

E = highest point in curve above which results are flagged as "E". The concentration range for target compounds is RL or MDL to E. Flag results <RL but >MDL as "J". Report result less than MDL as <RL.

MS/MSD Evaluation

If the concentration of a target analyte in the un-spiked (native) sample is more than four times the theoretical concentration of the matrix spike, the recovery is not reported and the data are flagged.

11.4 Identification "Tools"

Analysis by GC/MS (scan or SIM) may be used to confirm the presence of the target compounds (see SOP SM06: *Guidelines for SIM Analysis by GC/MS.)*

11.4.1 Relative Retention Time

The retention time of a surrogate compound or internal standard provides useful information about the stability of the GC system. If the surrogate RT has not changed, it is probable that the target analytes RTs have not changed. The relative retention time can help the analyst to evaluate a peak:

$$RRT = \frac{RT_{target}}{RT_{surrogate}}$$

The relative retention time will remain fairly constant under the same GC conditions. The expected retention time of the target can be estimated from the RRT and the RT of the reference (in this case, the surrogate):

$$RT_{target} = RRT \times RT_{surrogate}$$

The analyst must be alert for the presence of matrix interferences and evaluate the data on both columns before making an identification. Another useful tool that employs a similar idea to the RRT is to "overlay" the sample chromatogram with the calibration standard. If the chromatograms are scaled the same, the overlay provides good visual cues to the identification of the target compound.

11.4.2 Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the initial identification. The analyst is cautioned to use this approach with discretion and

with consultation with the GC supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a 2-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the GC supervisor or other manager with GC experience.

12. 0 QUALITY CONTROL AND DATA ASSESSMENT

12.1 The analytical batch is discussed in SOP AN02: *Analytical Batching and Evaluation of QC Data*, and these criteria are summarized in the SOP Summary included in Appendix C. Calculation of QC data is also given in SOP AN02.

13.0 METHOD PERFORMANCE

The Reporting Limits (RL), the Method Detection Limits (MDL), and accuracy and precision limits associated with these methods are given in the current revision of the Laboratory Quality Manual prepared by and for STL Savannah.

13.1 Initial and Continuing Demonstration of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP *CA92: Procedure for Initial and Continuing Analyst Demonstration of Capability.*

13.2 Method Detection Limit

The method detection limit must be determined for each analyte in accordance with SOP CA90: *Procedures for the Determination of Method Detection Limit (MDL).*

14.0 PREVENTIVE MAINTENANCE AND TROUBLESHOOTING

Refer to SOP AN53: *Maintenance Procedures for Laboratory Instrumentation* for routine preventive maintenance and the manufacturer's guides for trouble-shooting items.

15.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Follow the guidance for disposal in SOP CA70: *Waste Disposal.* Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Hexane extracts and hexane used to rinse glassware, columns, etc. Transfer to flammable waste containers.

16.0 REFERENCES

STL Savannah's *Laboratory Quality Manual (LQM)*, current revision

Severn Trent Laboratories' *Quality Management Plan (QMP), current revision*

Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846; including Update III. U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

Code of Federal Regulations, Title 40, Part 136; U.S. Government Printing Office: Washington, DC, July 1, 1988.

METHOD 515.1: DETERMINATION OF CHLORINATED ACIDS IN WATER BY GAS CHROMATOGRAPHY WITH AN ELECTRON CAPTURE DETECTOR Revision 4.1 Edited by J.W. Munch (1995) R.C. Dressman and J.J. Lichtenberg - EPA 600/4-81-053, Revision 1.0 (1981) J.W. Hodgeson - Method 515, Revision 2.0 (1986) D. J. Munch (USEPA, Office of Water) and T. Engel (Battelle Columbus Laboratories) - National Pesticide Survey Method 3, Revision 3.0 (1987) R.L. Graves - Method 515.1, Revision 4.0 (1989) **NATIONAL EXPOSURE RESEARCH LABORATORY OFFICE OF RESEARCH AND DEVELOPMENT U.S. ENVIRONMENTAL PROTECTION AGENCY CINCINNATI, OHIO 45268**

17.0 TABLES, DIAGRAMS, AND VALIDATION DATA

APPENDIX A – Target Compounds and Retention Times

Compound	RT COL 1	RT COL 2
Dalapon	2.18	2.17
DCAA (surrogate)	10.00	10.12
Dicamba	10.07	10.26
MCPP	10.30	10.31
MCPA	10.45	10.52
Dichlorprop	10.63	10.66
Bentazon	11.42	12.18
2,4-D	10.77	10.89
Pentachlorophenol	11.18	11.15
2,4,5-TP (Silvex)	11.28	11.30
2,4,5-T	11.50	11.62
2,4-DB	11.84	11.94
Dinoseb	11.93	11.87
Picloram	12.38	12.99
DCPA	12.52	12.66

COL1 _ J&W DB-XLB
COL2 _ J&W DB-35MS

Retention Times of Target Compounds with the following conditions:

Injector: 240°C
Detector: 300°C
Carrier Gas Flow: Helium at ~5.3mL/min (pressure at 20psi, constant)
Make-up Gas Flow: Nitrogen at ~60mL/min (per detector)

Temperature program:

Initial Temp:	50°C
Initial Hold:	0.50 min
Program Rate 1	12°C/min to 100°C
Program Rate 2	15°C/min to 200°C
Program Rate 3	80°C/min
Final Temp:	300°C (hold for 1.25 minutes)
TOTAL TIME	15.73 minutes

APPENDIX B - Example Standard Preparation Recipes

Intermediate Calibration Stock Standard

Stock Standard	Concentration (ug/mL)	Volume Added (uL) to final volume of 10mL in hexane	Final Concentration (ug/mL)
Herbicide Methyl Ester Mix	See below *	250	2.5/25/500
Pentachloroanisol	100	250	2.5
DCAA Methyl Ester	100	250	2.5
Picloram Methyl Ester	100	250	2.5
DCPA	1000	50	5.0

* Methyl Esters of MCPA/MCPP at 20000 ug/mL; Dalapon at 1000 ug/mL; 2,4-D, 2,4-DB, 2,4,5-T, 2,4,5-TP, Dicamba, Dichlorprop and Dinoseb at 100 ug/mL.

Calibration Standards

	Cal. Level 1*	Cal. Level 2*	Cal. Level 3*	Cal. Level 4**	Cal. Level 5*	Cal. Level 6*	Cal. Level 7*
Intermediate Calibration Stock Standard	100 uL	200 uL	400 uL	3000 uL	800 uL	1000 uL	2000 uL
Final Concentration	0.025/0.25/5.0 ug/mL	0.050/0.50/10 ug/mL	0.10/1.0/20 ug/mL	0.15/1.5/30 ug/mL	0.20/2.0/45 ug/mL	0.25/2.5/50 ug/mL	0.50/5.0/100 ug/mL

* 10 ml final volume in Hexane
** 50 mL final volume in Hexane

APPENDIX B - Example Standard Preparation Recipes

HERBICIDE MOLECULAR WEIGHTS AND CORRECTION FACTORS

Herbicide acid	MWacid	MWester/ether	Correction factor
2,4-D	221.04	235.07	0.940
Dalapon	142.97	157.00	0.911
2,4-DB	249.09	263.12	0.947
Dicamba	221.04	235.07	0.940
Dichloroprop	235.07	249.09	0.944
Dinsoeb	240.22	254.24	0.945
MCPA	200.62	214.65	0.935
MCPP	214.65	228.67	0.939
2,4,5-TP(Silvex)	269.51	283.54	0.951
2,4,5-T	255.48	269.51	0.948
DCAA	205.04	219.07	0.936
Picloram	241.48	255.51	0.945
Pentachlorophenol	266.35	280.37	0.950

Example Calculation

$$CF(2,4-D) = \frac{Wacid}{Wester} = \frac{221.04}{235.07} = 0.94$$

If the standard is expressed as mass of ester per volume, convert the concentration to he acid form by multiplying by the correction factor (CF).

APPENDIX C – Method Summary

HOLD TIMES

MATRIX (method)	Chemical Preservative/ Storage*	Routine Container	Sample Hold Time	Extract Hold Time
Drinking water (515.1)	80mg sodium thiosulfate per liter; 4°C	1-L amber x 2	14 days	28 days
Groundwater and Wastewater (615 and 8151)	None; 4°C	1-L amber x 2	7 days	40 days
Soils and wastes (8151)	None; 4°C	500-mL glass	14 days	40 days

*Storage temperature is 4°C with control criteria of less than 6°C with no frozen samples

EXTRACTION SUMMARY – detailed description in SOP EX45

Aqueous - adjust 500mL of sample to pH >12 and hydrolyze for one hour; extract with methylene chloride to remove non-targets and discard solvent; adjust aqueous phase to pH <=2 and extract with diethyl ether; concentrate, esterify and dilute to 10mL final volume with hexane

Soils - acidify 30g of sample, mix with acidified sodium sulfate; and sonicate with diethyl ether; hydrolyze with KOH at pH>12 and discard solvent; adjust aqueous phase pH to <=2 and extract with diethyl ether; concentrate, esterify, and dilute to final volume of 10mL with hexane.

ANALYSIS
Dual capillary columns with dual EC; 2-5uL injection into glass tee or y-splitter; external or internal standard calibration

SURROGATE:
DCAA - 2.0ug/L

BATCH QC
Method blank
LCS/LCSD- full target list of single peak analytes
MS/MSD- full target list of single peak analytes

Analytical Sequence

Method 515.1

Initial Calibration Standards
Initial Calibration Verification (ICV)
Laboratory Performance Solution (Daily)
Client samples analyzed until 12 hour clock expires
Calibration Verification standard – vary concentration
Client samples analyzed until 12 hour clock expires
Calibration Verification standard – vary concentration

Method 615

Initial Calibration Standards
Initial Calibration Verification (ICV)
Client samples analyzed until 24 hour clock expires
Calibration Verification standard – mid-level concentration
Client samples analyzed until 24 hour clock expires

Method 8151A

Initial Calibration Standards
Initial Calibration Verification (ICV)
20 client samples or 12 hours
Calibration Verification standard – mid-level concentration
20 client samples or 12 hours
Calibration Verification standard – mid-level concentration

The sequence continues until all samples have been analyzed or until the calibration verification fails the acceptance criteria. All sample extract analyses must be bracketed by acceptable verification standards if external standard calibration is used; if internal standard calibration is used, capping of the sequence by a CCV standard is not required unless specified in an agency or client QAPP. The default procedure is not to count the QC items in the 20 sample extracts that may be analyzed in the clock; i.e.; the number of sample and QC extracts may exceed 20 but the total number of sample extracts may not exceed 20 and all extracts (samples and QC) must be analyzed within the 12-hour clock.

Laboratory Performance Solution Criteria (EPA 515.1)

Test	Analyte(s)	Concentration (ug/mL)	Criteria
Sensitivity	Dinoseb	0.004	S/N >3

S/N = a ratio of peak signal to baseline noise.

EPA Method 515.1 requires the analysis of a Laboratory Performance Check (LPC). In addition to the sensitivity check for dinoseb, the EPA 515.1 LPC includes checks for chromatographic performance (using 4-Nitrophenol) and column performance (using 3,5-Dichlorobenzoic acid and 4-Nitrophenol). Since the laboratory does not report these analytes, only the dinoseb sensitivity check is required by this SOP.

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration- 5-point minimum with lowest point at RL	Prior to sample analysis or when CCV fails	1) 515.1 %RSD of each target <=20% or r^2 >0.99 2) 615 %RSD of each target <=10% or r^2 >0.99 3) 8151 %RSD of each target <=20% or r^2 >0.99 (see Section 10.2 for 8000-series "grand mean" exception)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and analyze standards
Initial calibration verification (Second Source ICV)	After Initial Calibration	515.1: Percent difference <=20% 615/8151: Percent difference <= 15%	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Laboratory Performance Check Solution	Daily, prior to sample analyses	See criteria in previous section.	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification (CCV)	515.1/8151: After every twenty sample analyses (or 12 hours) and at the end of the sequence 615: After every twenty sample analyses (or 24 hours)	515.1:Percent difference or drift <= 20% 615/8151: Percent difference or drift <= 15% (see Section 10.3 for 8000-series"grand mean" exception)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Method blank	Per batch	All targets reported less than RL in LQM	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze -Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze
Lab control sample (LCS)	Per batch	Recoveries within LQM limits	-Evaluate chromatogram and integrations. -Check calculations and reanalyze -Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix spike (MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. - Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze
Surrogate	All samples, method blanks, and QC	Recoveries within LQM limits	-Evaluate chromatogram and Integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze
Initial Demonstration of Capability (IDOC)	Initially and when new analysts trained	Evaluate in accordance with SOP 92 and method criteria	Repeat test for analytes that fail criteria
Method Detection Limit (MDL)	See SOP CA90	Evaluate in accordance with SOP CA90	Evaluate in accordance with SOP CA90
Retention time window determination	See SOP AN66	See SOP AN66	See SOP AN66

ORGANOCHLORINE PESTICIDES AND PCBs BY GC

(Methods: EPA 608, 8081A, and 8082)

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Technical Approval:

Andrea Teal 03/22/04
Date

Title: Quality Assurance Manager
STL Savannah

Safety Approval:

Ernest B. Watt 03/22/04
Date

Title: EHSC
STL Savannah

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures used to determine the concentration of chlorinated pesticides and polychlorinated biphenyls (PCBs) as Aroclors in various matrices. Appendix A contains an example of the retention time order for the single peak pesticides, Appendix B provides examples of the calibration standards routinely analyzed, and Appendix C contains a summary of the method QC requirements for Methods 608, 8081A, and 8082.

1.2 The routine target compounds, reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria are listed in the current revision of the Laboratory Quality Manual (LQM) prepared by and for STL Savannah.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 Environmental samples are prepared using matrix-specific procedures (see Section 9). The solvent is evaporated, the residue exchanged into hexane, and the sample adjusted to a final volume of 10mL or less. The preparation may also incorporate Florisil, copper (sulfur), acid (PCBs only), or gel permeation chromatography (GPC) cleanups. Analysis of the extract is routinely performed on a GC equipped with dual capillary columns (different phases) connected to dual electron capture (EC) detectors, allowing simultaneous detection and confirmation of the target compounds. GC/MS confirmation can also be employed if analyte concentration is sufficiently high or if the sample extract is concentrated to an appropriate final volume. Quantitation may be performed using the external or internal standards calibration technique.

2.2 Method Clarifications / Default Procedures

General Clarification: The procedures for chlorinated pesticides (8081A) and PCBs (8082) are given as separate methods in Update III of SW-846. In previous updates, pesticides and PCBs were included in a single method; pesticides and PCBs are still included in the scope of EPA Method 608. The extraction and the analysis are combined in this SOP 1) to reduce the time of extraction and analysis; and 2) to reduce the amount of solvent used in the procedures (one extraction instead of two). If interferences or high levels of non-PCB compounds are present, a portion of the extract can be subjected to the acid cleanup and reanalyzed. Note that if the list of target analytes includes only a limited list of components (i.e. Toxaphene, Chlordane, or PCBs), these procedures may be abbreviated to address only the analytes of interest.

Bracketing Sample Extracts: The laboratory's default procedure for continuing calibration verification stems from EPA Method 8000 and is to bracket samples by CCV standards (before and after) if external standard calibration is used and ***not*** to cap the sequence (run CCV after the samples) if internal standard calibration is used ***unless*** noted in an agency or client QAPP, in an STL pre-project plan, or in the method. EPA method 8081 specifically states to perform bracketing CCV every 12 hours (or 20 samples) and at the end of the analytical sequence; therefore, the requirement for capping CCV has been incorporated into this SOP.

Grand Mean: The "grand mean" is used to evaluate calibration data according to the provisions of SW-846 Method 8000B and Sections 10.3 and 10.4 of this SOP.

Quantitation of QC Items: The default procedure for the analysis and evaluation of QC items (method blank, LCS, and MS/MSD) is to analyze these items on one of the instruments used to analyze the associated samples.

Dilutions: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the

upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve. For example, a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client. Project managers and lab staff must work together to balance client satisfaction with productivity.

2.3 This method is based on the guidance in SW-846 Methods 8000B, 8081A, and 8082, and 40 CFR 136 Method 608.

2.4 Definitions – Refer to SOP AN99: *Definitions, Terms, and Acronyms* for a complete listing of applicable definitions.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.1 Specific Safety Concerns or Requirements

Acetone and hexane are flammable solvents. They can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

4.1 Glassware should be thoroughly cleaned and solvent-rinsed in accordance with SOP AN60: *Glassware Cleaning Procedures* to minimize artifacts and/or elevated baselines in gas chromatograms. Any vessel that comes in contact with the extract is a potential source for contamination. Method blanks that are extracted and analyzed with each batch of samples will provide clues to the source of contamination from the glassware and reagents.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. See Section 9 for a table summary of the cleanups that may be employed to eliminate or reduce interferences. If matrix interferences continue after a cleanup has been performed, the sample is diluted as needed for data analysis. If a cleanup is used, the method blank and laboratory control standard must also be subjected to the cleanup.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Aqueous samples are collected in 1-L glass containers with Teflon-lined caps. Soil/sediment samples are collected in wide mouth glass jars equipped with Teflon lined caps. No preservative is added. The samples are iced at the time of collection and maintained at 4°C (less than 6°C with no frozen samples) until extraction. Extraction must be performed within 7 days for aqueous samples and within 14 days of sampling for soils/solids. The extracts must be stored at 4°C (less than 6°C) and must be analyzed within 40 days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph (GC), temperature programmable, equipped with single or dual electron capture (EC) detectors and a compatible autosampler

6.2 Data system compatible with the GC, with appropriate software or integration capabilities

6.3 The following column pairs are recommended. Other columns/phases may be used if the calibration and QC criteria are met and adequate separation of the target compounds is achieved.

CLP I fused silica capillary column 30 M x 0.53 mm ID x 1.5 µm film
CLP II fused silica capillary column 30 M x 0.53 mm ID x 0.83 µm film

6.4 Microsyringes, appropriate volumes

6.5 Volumetric flasks, Class A, appropriate volumes

6.6 Autosampler vials, septa, and caps - compatible with the autosampler

7.0 REAGENTS

All reagents must be tracked in accordance with SOP AN44: *Reagent Traceability.*

Hexane - pesticide grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SOP AN41: *Standard Material Traceability.* General guidance on the preparation of standards is given in SOP AN43: *Standard Preparation.*

The lab should purchase certified solutions from STL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SOP AN43 for guidance for standard preparation.

Calibration Standard Recipes

The recipes used for standard preparation must be clearly documented as a controlled posting or as a narrative in the traceability log. The lowest level calibration standard should be at or below the equivalent of the reporting limit as defined in the LQM or client QAPP. The remaining standards will define the working range of the analytical system. Appendix B contains example recipes of the calibration levels for the routinely determined single peak pesticides, technical chlordane, toxaphene, and the Aroclors.

If internal standard calibration is used, each calibration standard must contain the same concentration of the internal standard(s). The recommended concentration range for the internal standard(s) is 0.050 to 0.10µg/mL.

9.0 SAMPLE PREPARATION

The sample preparation and cleanup procedures are described in the following SOPs:

PROCEDURE	MATRIX	STL SOP
Continuous Liquid-Liquid extraction	aqueous and leachates	EX30
Ultrasonic extraction	soils and sediments	EX40
Waste dilution	Waste samples (oils, products, etc)	EX42
Zymark extract concentration	All extracts	EX50

CLEAN-UP PROCEDURE	STL SOP	APPLICATION	EFFECTIVENESS
Florisil	EX62	Pest/PCBs	Eliminates polar non-target compounds
Sulfuric acid	EX60	PCBs	Eliminates some unsaturated hydrocarbon interferences
Copper	EX60	Pest/PCBs	Eliminates elemental sulfur
GPC	EX61	Pest/PCBs	Eliminates high molecular weight non-target compounds and sulfur

10.0 ANALYTICAL PROCEDURE

10.1 Gas Chromatograph Operating Conditions
The instrument conditions listed in this section are for guidance. The actual conditions used by the lab must be documented in the instrument maintenance log, data system, or run log. The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

10.1.1 Two configurations are routinely used for the analysis of pesticides and PCBs. A single column may be connected to the injection port or two columns may be connected to the injection port using a press-tight glass y-splitter and a guard column, a two-hole ferrule, or a glass tee to provide simultaneous detection and confirmation of the target analytes.

10.1.2 Example GC Parameters

Injector: 220 – 240°C
Detector: 300 – 320°C
Carrier Gas Flow: Helium at 5 mL/min (per column)
Make-up Gas Flow: Nitrogen at 25 mL/min (per detector)-see manufacturer's recommended flows

Example chromatogram temperature program:

Initial Temp:	160°C
Initial Hold:	4.0 min
Program Rate:	10°C/min
Final Temp:	270°C (hold for 10 minutes)
Injected Volume:	2-4µL - 1-2µL per column (single injection into guard column and "Y" splitter)

NOTE: These conditions and parameters are given for guidance. The columns/phases, GC conditions, and instrument parameters may be modified to optimize each analytical system.

10.2 Column Evaluation (608 and 8081A)

10.2.1 PEVAL Breakdown Standard

The column(s) must be evaluated prior to the analysis of the calibration standards and once every 12 hour clock. (NOTE: For EPA 608, this column evaluation is required once every 24 clock.) The column evaluation is performed by injecting a PEVAL standard that contains Endrin and p,p'-DDT and calculating the percent breakdown of these compounds. The standard used for determining the percent breakdown must not contain any compounds that coelute with Endrin, DDT, or any of the corresponding breakdown products.

NOTE: This column evaluation does not have to be performed if PCBs only are the target compounds. PCBs are stable and not subject to breakdown in the injection port.

Inject the Endrin/DDT breakdown standard. Check the peak integrations and calculate the breakdown as follows:

$$\%\text{Breakdown Endrin} = \frac{\text{Response(Endrin Aldehyde + Endrin Ketone)}}{\text{Response(Endrin + Endrin aldehyde + Endrin Ketone)}} \otimes 100$$

$$\%\text{Breakdown DDT} = \frac{\text{Response(DDE + DDD)}}{\text{Response(DDT + DDE + DDD)}} \otimes 100$$

The response (area or height) must be used to evaluate the breakdown. Do not use concentrations and do not "undetect" peaks that are below the RL or MDL. All peaks detected by the data system must be included in the percent breakdown calculation.

Breakdown Criterion
The breakdown for each compound must be less than 15%. If the breakdown exceeds 15%, the instrument will require column and/or injector port maintenance. The maintenance may include but is not limited to replacing the septum, clipping the front of the guard column, replacing the glass injector sleeve, and scrubbing (cleaning) the injector port.

10.2.2 If the instrument has not been in use for more than one day, a "priming" analysis may be beneficial. The analysis of a relatively high concentration pesticide or PCB standard may help to stabilize the response of the very sensitive EC detector. Inject a standard that is about 10x the concentration of the highest calibration standard and allow the instrument to cycle through the temperature program. It is not necessary to acquire the data but the baseline should be monitored before and after the priming analysis to gauge the condition of the detector. A hexane blank should be analyzed after the analysis of the priming standard and before the % breakdown check.

NOTE: The "priming" standard should be injected manually to avoid contaminating the autosampler syringe.

10.3 Initial Calibration

Initital calibration must be performed in accordance with SOP AN67: *Evaluation of Calibration Curves*. Internal or external standard calibration techniques may be employed for the determination of the concentration of pesticides and PCBs. Pentachloronitrobenzene (PCNB) or 2-Nitro-1-bromobenzene is recommended for use as internal standards; however, other compounds may be used.

Internal standard calibration should be used as the default. If matrix interferences preclude the use of internal calibration for a sample extract, two options should be considered:

1) dilute the extract or perform sample extract cleanup to minimize or eliminate the interference
2) use external standard calibration to quantify the target and surrogate compounds (if external standard calibration is used, all calibration requirements, including a capping standard, must be met - see Appendix C for the external standard sequence).

10.3.1 Prepare and analyze the calibration standards. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards. Guidance for establishing the analytical sequence is given in the SOP Summary.

Note that the following offers two options for calibration and quantitation – average CF or regression curve. Only one needs be chosen per analyte.

10.3.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the hexane blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed? Is the column properly seated in the injector and detector ports?
>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?
>Are the peaks symmetrical? Is there tailing or fronting?
>Are the areas of the peaks normal for the sensitivity setting being used?

Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

10.3.3 Evaluate the calibration curve in accordance with SOP AN67: *Evaluation of Calibration Curves*.

10.3.4 Initial Calibration Criteria:

600-series: If the relative standard deviation is less than 10% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

8000-series: If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

The preferred method of quantitation is the average response or calibration factor. If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve. The "grand mean exception" described below should be applied to 8081A and 8082 initial calibrations only in extraordinary circumstances because of the difficulty of maintaining and providing documentation on an on-going basis.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average calibration factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%, with no individual compound exceeding 3X the ICAL criteria (60%).
NOTE: If a target compound that passes by the "grand mean exception" is detected the PM is notified via an anomaly report or case narrative.

Regression Curve Option: A calibration curve is established for each analyte by plotting the concentration along the x-axis and the corresponding response along the y-axis. If the regression coefficient of the regression curve is greater than 0.99, the curve can be used to quantify

samples. For 8000-series methods, a minimum of five points is required for a linear regression, six points for a second order (quadratic) curve, and seven or more for higher order fits. It is recommended to use only linear and quadratic (second order) curves for quantitation. See SOP AN67 for guidance on evaluation of calibration curves.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

10.4 Calibration Verification

A second source initial calibration verification standard must be performed in accordance with SOP AN67.

Calibration is verified at the frequency given in the SOP Summary. The following criteria apply to calibration standards analyzed before and after samples. In situations where compounds fail criteria high and no positive hits for the compound(s) failing high are detected, these samples may be reported if appropriately qualified.

Analyze a mid-level standard. The concentration of the verification standard should be varied quarterly to evaluate the calibration curve in the lower and upper halves. Tabulate the area of the target analytes and calculate the response factors if using the average RF/CF option. If using the calibration curve option, calculation of the RF is unnecessary.

Calculate the percent drift or percent difference between the initial and continuing calibration in accordance with SOP AN67.

10.4.2 Continuing Calibration Verification Criteria

Response Criteria
If the CCV criterion is not met, another CCV should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the calibration verification criteria cannot be met, a new initial calibration must be prepared, analyzed, and evaluated.

600-series: If the percent drift or percent difference is less than or equal to 15%, the initial calibration is verified and the average response factor or regression curve can be used for quantitation.

8000-series: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor or regression curve can be used for quantitation.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average calibration factor or regression curve from the initial calibration can be used for quantitation if the average %drift or average % difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15% and no single compounds exceeds 3X (45%) the CCV criteria.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative.

All samples must be bracketed by acceptable CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no

target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (per SW-846 Method 8000B).

Retention Time Criteria
The retention time for the CCV must fall within the daily retention time window as defined in SOP AN66: *Determination of Retention Time Windows for Gas Chromatographic Analyses.*

Internal Standard Response Criteria
If internal standard calibration is used, the response of the internal standard(s) must be within –50% to +150% of the response in the CCV-level standard in the initial calibration sequence. If the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

10.5 Sample Analysis Sequence
The analytical sequences for the 600- and 8000-series methods are given in the SOP Summary in Appendix C. The default is to exclude QC items (method blanks, LCS, and MS/MSD) in determining the maximum number of extracts in the clock.

For 8081 and 8082, more than 20 extracts (samples and QC) may be analyzed in a sequence, as long as the 12 hour time frame has not elapsed, but the number of samples (non-QC extracts) may not exceed 20. Note that some client and agency QAPPs may require that the QC items be counted as part of the twenty samples.

For 608, more than 20 extracts (samples and QC) may be analyzed in a sequence, as long as the 24 hour time frame has not elapsed, but the number of samples (non-QC extracts) may not exceed 20. Note that some client and agency QAPPs may require that the QC items be counted as part of the twenty samples.

10.5.1 The sample extract is injected using the same injection volume used for the calibration standards. Extracts that are known to be relatively clean should be analyzed first. Extracts suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

If the internal standard calibration is used, the concentration(s) of the internal standard(s) must be the same in all calibration samples, field samples, and QC samples. A concentration of 0.050ug/mL to 0.10ug/mL (final extract concentration) is recommended.

10.5.2 If the concentration of target compounds exceeds the working range (defined by the highest standard in the initial calibration), the extract must be diluted in hexane and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve. If the internal standard calibration is used, the concentration of the internal standard in the diluted extract must be the same as in the calibration standards.

NOTE: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve. For example, a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to

provide lower detection limits to the client. Project managers and lab staff must work together to balance client satisfaction with productivity.

10.5.3 Occasionally, situations may arise where part of the chromatogram is obscured by large non-target peaks (such as phthalate esters, which elute in the same general retention time range as the pesticides and PCBs) or matrix interferences (short, wide, peaks that are not well resolved). In these situations, it is permitted to report a lower RL for the target compounds that are not affected by the non-target or matrix interference and perform a dilution only for the target compounds that are affected. This anomalous situation must be discussed with the project manager and section supervisor prior to reporting the results and noted in the case narrative or anomaly report. Again, project managers and lab staff must work together to balance client satisfaction with productivity.

10.6 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SOP AN66: *Determination of Retention Time Windows and Evaluation of Retention Time Data for Chromatographic Analyses.* If internal standard calibration is used relative retention times, as described in Section 11.1.4, are used to identify the target compounds.

11.0 DATA ANALYSIS AND CALCULATIONS

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times); the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration; and the integration of the peaks. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data. SOP AN66: *Determination of Retention Time Windows and Evaluation of Retention Time Data for Chromatographic Analyses* provides additional guidance for the evaluation of chromatographic data. This guidance is summarized in the following sections.

Manual integrations must be documented in accordance with SOP AN65: *Manual Integrations.* Data systems should be adjusted to minimize operator intervention. All chromatographic peaks must be evaluated for overall peak shape and "reasonableness" of integration. Under no circumstances should manual integrations be used to change reasonable data system integrations in order to meet calibration or QC criteria.

The judgement and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks properly integrated?
- Are co-eluting peaks or matrix interferences present?
- Is the internal standard present at the correct retention time and response (-50% to150% of the response in the associated CCV)? Are the surrogates present at the expected RT or have they shifted?

11.1 Qualitative analysis

Identification of the surrogates and target compounds is based on retention time. The retention time (RT) windows calculated around the CCV retention times are used for the identification of the target compounds. The analyst should also note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect". This may only be done when the RT of the internal standard and surrogates are at their respective retention times and there is little or no evidence of matrix interferences. If there is doubt as to whether the peak can be excluded or not, the default procedure will be to report the peak as the target compound unless another technique (for example, GC/MS) is used to determine that the target compound is not present.

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peak areas that exceed the established linear range of the calibration curve may result in significant retention time shifts; therefore, all peaks, which have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are handled using dilutions as detailed previously.

11.1.1 Evaluate the internal standard (if used) and the surrogates to check for shifts in retention times and to evaluate the surrogate recovery. The recovery criteria for surrogates are given in the LQM.

Internal Standard Criteria
The internal standard must be within the retention time window defined by the associated CCV. The response of the internal standard(s) must be within a range ±50% of the response of the internal standard in the associated CCV.

If sample matrix interferences preclude the use of internal calibration for a sample extract, two options should be considered:
1) dilute the extract or perform sample extract cleanup to minimize or eliminate the interference
2) use external standard calibration to quantify the target and surrogate compounds (if external standard calibration is used, all calibration requirements, including a capping standard, must be met - see Appendix C for the external standard sequence).

Surrogate Criteria
A minimum of two surrogates is spiked into each sample and QC item prior to preparation. Decachlorobiphenyl (DCB) and 2,3,4,6-tetrachloro-m-xylene (TCMX) are the recommended surrogates. DCB should be evaluated as the primary surrogate; TCMX is evaluated if there is matrix interference with DCB.

NOTE: TCMX (or an alternate surrogate such as octachloronaphthalene) is to be used to evaluate data when AR1268 is detected. That is, TCMX or the alternate surrogate must pass the surrogate recovery criterion in order to report the data without qualification. AR1268 contains DCB and will bias the recovery when DCB is evaluated as a surrogate.

Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Evaluate the surrogates in the same manner as the target compounds using the guidance in the table in Section 11.1.3.

NOTE: If the recovery of the surrogate(s) is above the upper control limit and no target compounds are detected in the sample, results may be reported with appropriate qualification. Refer to SOP AN02: *Analytical Batching and Evaluation of QC Data* regarding this issue.

Dilutions and Surrogate Recovery
The concentration range for surrogates is approximately 0.0025µg/mL to 0.080µg/mL. This should give up to a six-fold dilution to report surrogate recoveries, if the spiking level is 0.5µg/L and the lower limit of quantitation is 0.025µg/L. The lower recovery limit is 30%; therefore, the lowest acceptable concentration is 0.5*0.3=0.15µg/L. The highest dilution to report un-qualified results ("J" flag) would be 0.15µg/L/0.025µg/L = 6 or a six fold dilution.

11.1.2 Evaluate each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences, and the overall shapes of the peaks.

If the concentration is below the reporting limit standard or MDL (if the sample is being evaluated for "J" results), the reporting limit (RL) for that compound is calculated (Section 11.2). The RL is calculated for all target compounds that are not detected on the primary analytical column. Peaks over-range are handled using dilutions as detailed above (10.5.2).

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected on the confirmation column or if the concentration is within the calibration range with the %RPD >40, the analysis at a dilution is not necessary.

11.1.3 If the result for a target is above the reporting limit (RL) on the primary column, evaluate the confirmation column. Use the retention time window calculated using the CCV as guidance for the identification of the target compounds. Note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect".

If the target compound is detected on the confirmation column, the concentration of the target compound is calculated and compared to the result from the primary column. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

If the relative percent difference is less than or equal to 40%, the presence of the target compound is confirmed and the higher concentration is reported.

NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A larger relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, the analyte is confirmed; however, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. Report the result that is most reasonable for the sample. The lower result is reported as the default when the %RPD is >40. Flag the result to note the disparity (P flag) between the columns.

NOTE: The default guidance instructs the analyst to report the highest result when the %RPD <=40% and to report the lowest result when the %RPD exceeds 40%. The guidance uses RPD to determine if matrix interference is present. If the RPD is <=40%, the results between the two columns are essentially the same, and no matrix interference is deemed present. If the RPD exceeds 40%, a matrix interference is most likely present, and the lower result is reported as the default. This guidance does not prohibit reporting the higher result when the %RPD exceeds 40; however, this guidance is intended to preclude reporting clearly unreasonable data. It is this laboratory's experience that a majority of matrix interferences are positive and that reporting the lower of the two results is reasonable when the %RPD exceeds 40.

Alternatively, perform additional extract cleanup (sulfur, florisil, etc.) or if cleanup is not feasible or there is a low probability that cleanup will help, dilute the extract to a level that removes the interference and report the RL from this dilution.

The table below gives default guidance for evaluating hits.

The default guidance in this table assumes the following:
1) the retention time and response of the internal standard(s) are within acceptance criteria with little or no shift in RT
2) surrogate recoveries meet the acceptance criteria and peaks fall within the middle of the retention time window with little or no shift in RT
3) the peak identified as the target falls in the middle of the retention time window for that compound

Default Guidance for Evaluation of Surrogates and Target Compounds in Samples, LCS, and MS

PEAK INFORMATION	COLUMN 1	COLUMN 2	%RPD	REPORT
No peak present	No peak		NA	<RL
		No peak	NA	If compound is a surrogate, re-extract. If sample is LCS, re-extract.
Peak present at RT	<E	<E	<=40%	Report highest
	<E	<E	>40%	Report result most appropriate for sample matrix. Use lowest result as default. Flag with "P"
Peak present at RT	>E	<E	<=40%	Dilute extract to get both results within the calibration curve.
	<E	>E		
	>E	<E	>40%	Report lowest result and flag with "P" No dilution required.
	<E	>E		

E = highest point in curve above which results are flagged as "E". The concentration range for target compounds is RL or MDL to E. Flag results <RL but >MDL as "J". Report results less than MDL as <RL.

MS/MSD Evaluation
If the concentration of a target analyte in the un-spiked (native) sample is more than four times the theoretical concentration of the matrix spike, the recovery is not reported and the data is flagged.

11.1.5 Identification "Tools"
Analysis by GC/MS (scan or SIM) may be used to confirm the presence of the target compounds in accordance with SOP SM06: *Guidelines for SIM Analysis by GC/MS.*

11.1.5.1 Relative Retention Time

The retention time of a surrogate compound or internal standard provides useful information about the stability of the GC system. If the surrogate RT has not changed, it is probable that the target analytes RTs have not changed. The relative retention time can help the analyst to evaluate a peak:

$$RRT = \frac{RT_{target}}{RT_{surrogate}}$$

The relative retention time will remain fairly constant under the same GC conditions. The expected retention time of the target can be estimated from the RRT and the RT of the reference (in this case, the surrogate):

$$RT_{target} = RRT \times RT_{surrogate}$$

The analyst must be alert for the presence of matrix interferences and evaluate the data on both columns before making an identification. Another useful tool that employs a similar idea to the RRT is to "overlay" the sample chromatogram with the calibration standard. If the chromatograms are scaled the same, the overlay provides good visual cues to the identification of the target compound.

11.1.5.2Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the initial identification. The analyst is cautioned to use this approach with discretion and with consultation with the GC supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a 2-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the GC supervisor or other manager with GC experience.

11.1.6 Qualitative Analysis of Multiple Peak Compounds

Identification of multi-peak pesticides (Toxaphene and Technical Chlordane) and PCBs as Aroclors is based on the recognition of their chromatographic patterns. Quantitation is performed using the area of characteristic peaks in the sample and standard using external or internal calibration procedures.

If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be warranted. Suggested cleanup options are given in Section 9.

11.1.6.1PCBs as Aroclors

PCBs are generally reported as Aroclors. The Aroclors have varying levels of PCB congeners with the last two numbers in the Aroclor designation indicating the weight percent of chlorine. For example, AR1221 is 21% chlorine by weight; AR1260 is 60% chlorine by weight. The 12- in the Aroclor designation represents the biphenyl molecule. The exception to this naming convention is AR1016, which is about 42% chlorine by weight. (Note that AR1016 and AR1242 have similar chromatograms - both Aroclors have almost the same weight of chlorine by weight and nearly the same PCB congeners.)

Aroclors are identified by matching the pattern of the sample with standards analyzed under the same analytical conditions. Interference may occur due to the presence of non-target analytes or due to "weathering" of the Aroclor in the environment. The presence of multiple Aroclors will also complicate identification and quantitation. Many matrix interferences may be reduced or eliminated by treating the sample extract with copper, and sulfuric acid, prior to analysis. STL SL SOP EX60 details this procedure.

NOTE: Do not use the acid cleanup on the entire extract if pesticides are also to be reported as many of the pesticides are not stable in acid or strong oxidizer.

When a pattern matching an Aroclor is encountered, it may be quantitated using either the 3-5 characteristic peaks (recommended) or total area response. Total area quantitation should only be used as detailed below. Residues of either AR1016 or AR1260 are quantitated using the average RF/CF determined during initial calibration. The other Aroclors are quantitated against the RF/CF determined from their single-point analysis during initial calibration. Samples should be diluted when the amount of PCB in a sample extract exceeds the calibration range defined in initial calibration. Note that the AR1660 standard defines the working range for all the Aroclors. (i.e. if AR1660 was calibrated from 0.10µg/mL to 5.0µg/ml, and a sample extract was analyzed containing 10µg/ml of AR1232, that extract would require dilution to get the amount of AR1232 to be less than 5.0µg/ml.) If a sample contains any of the single point Aroclors (that is, Aroclors other than AR1016 and AR1260), then that associated standard must be run within 72 hours of the sample to determine retention time shifts and pattern recognition.

In the 3-5 peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of PCB in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 3-5 resulting concentrations are averaged to provide the final result for the sample.

"Weathering" is the loss of part of the Aroclor pattern due to biological or chemical degradation of individual PCBs. When weathering is suspected, try to match the later eluting peaks first. Flag the results for a weathered Aroclor pattern as tentatively identified or make a note in the case narrative if provided.

The presence of multiple Aroclors can be a problem to identify since most Aroclors have at least a few peaks in common. The easiest case would be to have early and late eluting Aroclors present. The most difficult cases will involve the presence of Aroclors with the same relative chlorine level.

NOTE: When choosing individual peaks for quantitation, compare their responses in the sample and standard. If the peaks chosen do not correlate well (i.e. ratios to other peaks are close) between the sample and standard, review the chromatograms for other possible peaks for quantitation.

11.1.5.2 Toxaphene

Toxaphene is a mixture of chlorinated camphenes, which has a complex and characteristic pattern when analyzed by GC-ECD. A single Toxaphene standard is analyzed during the initial calibration for the purpose of pattern identification in samples. When a Toxaphene residue is detected in sample(s), sample analysis is stopped. A calibration curve with at minimum of 5 points bracketing the instrument calibration range for Toxaphene should be analyzed. Alternatively, single points may be prepared with Toxaphene concentrations within 2x the Toxaphene quantity detected in the samples. Generally, the calibration curve option is simpler. After analysis of the Toxaphene standard(s), the samples are re-analyzed using this standard(s) for quantitation. Note that when analysis of Toxaphene-containing samples occurs over an

extended time, the calibration factor should be verified or regenerated every 12 hours for method 8081, and every 24 hours for method 608.

If the sample and standard chromatograms agree well, Toxaphene is quantitated using 5 characteristic peaks (similar to the PCB approach, above).

When Toxaphene is determined using the 5-peak approach, the analyst must take care to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms. It is highly unlikely that the peaks will match exactly, but the analyst should not employ peaks from the sample chromatogram whose relative sizes or areas appear to be disproportionately larger or smaller in the sample compared to the standard.

In the 5-peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of Toxaphene in the standard. A minimum of three peaks must be used to determine the calibration factor. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 5 resulting concentrations are averaged to provide the final result for the sample.

11.1.5.3 Chlordane

Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. The following components are significant: α and γ Chlordane, trans-Nonachlor, Heptachlor, and Heptachlor-epoxide. The α and γ Chlordane isomers are the most prevalent and their detection as single components is a good indicator that Technical Chlordane may be present.

When the GC pattern of the residue resembles that of the Technical Chlordane standard, quantitate Chlordane residues by comparing the area of 3 to 5 major peaks. Heptachlor and heptachlor epoxide should not be included in this quantitation but rather should be quantitated and reported separately.

When a Technical Chlordane residue is detected in sample(s), sample analysis is stopped. A calibration curve with at minimum of 5 points bracketing the instrument calibration range for technical chlordane should be analyzed. Alternatively, single points may be prepared with technical chlordane concentrations within 2x the Technical Chlordane quantity detected in the samples. Generally, the calibration curve option is simpler. After analysis of the technical chlordane standard(s), the samples are re-analyzed using these standard(s) for quantitation. Note that when analysis of technical chlordane-containing samples occurs over an extended time, the calibration factor should be verified or regenerated every 12 hours for method 8081, and every 24 hours for method 608.

NOTE: These procedures are not necessary if the lab is reporting chlordane as the α and γ chlordane isomers, not as the technical product.

11.2 Refer to SOP AN67: *Evaluation of Calibration Curves* for the calculations of sample concentrations.

12.0 QUALITY CONTROL AND DATA ASSESSMENT

12.1 Analytical Batching

QC data must be evaluated against the precision and accuracy criteria set forth in the Laboratory Quality Manual and SOP AN02: *Analytical Batching and Evaluation of QC Data*. These criteria are summarized in the SOP Summary included in Appendix C

SOP AN02 also provides guidance for establishing and evaluating QC items to be included in an analytical batch.

12.2 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP CA85: *Nonconformance and Corrective Action Procedures.* CA85 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures.

13.0 METHOD PERFORMANCE

The Reporting Limits (RL), the Method Detection Limits (MDL), and accuracy and precision limits associated with these methods are given in the current revision of the Laboratory Quality Manual prepared by and for STL Savannah.

13.1 Initial and Continuing Demonstration of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP *CA92: Procedure for Initial and Continuing Analyst Demonstration of Capability.*

13.2 Method Detection Limit

The method detection limit must be determined annually for each analyte in accordance with SOP CA90: *Procedures for the Determination of Method Detection Limit (MDL).*

14.0 PREVENTIVE MAINTENANCE AND TROUBLESHOOTING

Refer to SOP AN53: *Maintenance Procedures for Laboratory Instrumentation* for routine preventive maintenance and the manufacturer's guides for trouble-shooting items.

15.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15.1 Waste Streams Produced by the Method

Excess samples, reagents, and standards must be disposed in accordance with SOP CA70: *Waste Management.*

The following waste streams are produced when this method is carried out:

- Flammable waste (hexane and methanol from extracts, rinsings, and standards) – Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full.
- Excess water samples. Dispose according to characterization on sample disposal sheets. If non-hazardous, dispose down drain/sewer. If hazardous, transfer to hazardous waste department for storage.
- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.
- Methylene chloride extracts. Dispose according to characterization on sample disposal sheets. If non-hazardous, transfer extract to chlorinated waste container. If hazardous, transfer to hazardous waste department for storage.

16.0 REFERENCES

Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846; including Update III. U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

Code of Federal Regulations, Title 40, Part 136; U.S. Government Printing Office: Washington, DC, July 1, 1988.

17.0 TABLES, DIAGRAMS, AND VALIDATION DATA

Appendix A: CHLORINATED PESTICIDES AND PCBs STANDARDS

Routine Targets

Target compound	STD ISMA-1 (ug/mL)	STD ISMA-2 (ug/mL)	STD ISMA-3 (ug/mL)	STD ISMA-4 (ug/mL)	STD ISMA-5 (ug/mL)
TCMX, DCB, OCN (surr)	0.0025	0.0050	0.010	0.020	0.040
g-BHC(Lindane), Heptachlor, Heptachlor epoxide, Endosulfan I, a-BHC, b-BHC, d-BHC, a-Chlordane, g-Chlordane	0.0050	0.010	0.020	0.030	0.050
Dieldrin, p,p'-DDT, Endosulfan II, Endrin aldehyde, Methoxychlor, p,p'-DDE, Endrin p,p'-DDD, Endosulfan sulfate, Endrin ketone	0.010	0.020	0.040	0.060	0.10

DDT/Endrin Breakdown Evaluation Standard

Pesticide Evaluation Standard	CONC (ug/mL)
Endrin,	0.040
P,P'-DDT	0.040

Appendix IX Targets

Target compound	APIX-1 (ug/mL)	APIX -2 (ug/mL)	APIX -3 (ug/mL)	APIX -4 (ug/mL)	APIX -5 (ug/mL)
TCMX, DCB, OCN (surr)	0.0025	0.0050	0.010	0.020	0.040
Isodrin	0.0050	0.010	0.020	0.030	0.050
Chlorobenzilate	0.050	0.10	0.20	0.50	1.0
Kepone	0.025	0.050	0.10	0.20	0.50

APPENDIX B

Technical Chlordane Five-point Curve

STOCK STANDARD	TCHLOR -1	TCHLOR -2	TCHLOR -3	TCHLOR -4	TCHLOR -5
Technical Chlordane	0.10	0.20	0.40	0.60	0.80
TCMX, DCB OCN (surr)	0.0025	0.0050	0.010	0.020	0.040

Toxaphene Five-point Curve

STOCK STANDARD	TOX -1	TOX -2	TOX -3	TOX -4	TOX -5
Toxaphene	0.10	0.20	0.40	1.0	2.0
TCMX, DCB, OCN (surr)	0.0025	0.0050	0.010	0.020	0.040

PCBs as Aroclors

AR1660 Standards

Calibration Std	AR1016 (ug/mL)	AR1260 (ug/mL)	Surrogates (ug/mL)
AR1660-1	0.10	0.10	0.0025
AR1660-2	0.20	0.20	0.0050
AR1660-3	0.50	0.50	0.010
AR1660-4	1.0	1.0	0.020
AR1660-5	2.0	2.0	0.040

Single Point Aroclor Calibration Standards

Calibration Standard	Single Pont Concentration (ug/mL)	Surrogate Concentrations (ug/mL)
AR1221	1.0	0.020
AR1232	1.0	0.020
AR1242	1.0	0.020
AR1248	1.0	0.020
AR1254	1.0	0.020

If required, five point curves for AR1221, AR1232, AR1242, AR1248, and AR1254 are prepared at the same concentrations as the AR1660 curve.

Appendix B - METHOD SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage*	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	None; 4C	1-L amber	7 days	40 days
Soil/ Sediment	None; 4C	500-mL	14 days	40 days
Waste	None; 4C	Glass	14 days	40 days
TCLP	None; 4C	1-L amber	7 days to leach / 7 days (after leaching procedure)	40 days

*Storage temperature is 4°C with control criteria of less than 6°C with no frozen samples

EXTRACTION

Aqueous: Approximately 1L of sample (contents of container) using continuous at pH 5-9 with methylene chloride; exchange to hexane and concentrate to final volume of 10mL

Soil/Solids: Approximately 30g of sample using sonication with 1:1 acetone/hexane or 1:1acetone/methylene chloride; Concentrate to final volume of 10mL in hexane

Wastes: Approximately 1g of sample diluted to final volume of 10mL with hexane

ANALYSIS

Dual capillary columns with dual EC; 2-5uL injection into glass tee or y-splitter; external or internal standard calibration

SURROGATE:	Aqueous (ug/L)	Soils (ug/kg)
Tetrachloro-m-xylene	0.50	15
Decachlorobiphenyl	0.50	15
OCN	0.50	15

608 BATCH QC

Method blank
LCS/LCSD- full target list of single peak analytes @ 0.20ug/L
MS/MSD- full target list of single peak analytes @ 0.20ug/L

8081/8082 BATCH QC

Method blank
LCS - LQM subset
MS/MSD - LQM subset

Parameter	Aqueous (ug/L)	Soils (ug/kg)
Lindane	0.20	6.0
Aldrin	0.20	6.0
Heptachlor	0.20	6.0
Dieldrin	0.50	15
Endrin	0.50	15
p,p'-DDT	0.50	15

Appendix C

The sequence continues until all samples have been analyzed or until the calibration verification fails the acceptance criteria. All sample extract analyses must be bracketed by acceptable verification standards. The default procedure is not to count the QC items in the 20 sample extracts that may be analyzed in the clock; i.e.; the number of sample and QC extracts may exceed 20 but the total number of sample extracts may not exceed 20 and all extracts (samples and QC) must be analyzed within the 12-hour clock for 8081/8082 and a 24-hour clock for 608.

ANALYTICAL SEQUENCE – 608, 8081A, and 8082 (Pesticides and PCBs)

STANDARD / SAMPLES
Initial Calibration - 5 point single peak pesticides 5 point AR1660 (*Note 2) 1 point Toxaphene (*Note 1) 1 point Technical Chlordane (*Note 1) 1 point remaining Aroclors
Instrument Blank
PEVAL - Endrin/p,p'-DDT Breakdown Check (*Note 3)
ICV (Initial Calibration Verification) - 2nd source calibration verification
Up to twenty sample extracts and QC and/or 12 hours for 8000-series Up to twenty sample extracts and QC and/or 24 hours for 600-series
PEVAL - Endrin/p,p'-DDT Breakdown Check (*Note 3)
CCV (Continuing Calibration Verification) - Midpoint single peak pesticides and midpoint AR1660 (*Note 2)
Instrument Blank
Up to twenty sample extracts and QC and/or 12 hours for 8000-series Up to twenty sample extracts and QC and/or 24 hours for 600-series
PEVAL - Endrin/p,p'-DDT Breakdown Check (*Note 3)
CCV (Continuing Calibration Verification) - Midpoint single peak pesticides and midpoint AR1660 (*Note 2)
Instrument Blank

*Note 1 – If Toxaphene or Technical Chlordane is detected in a sample, a full 5-point ICAL must be performed and the sample reanalyzed.

*Note 2 - A mixture of AR1016 and AR1260 will be used to calibrate and verify the response for PCBs. Mid-level standards of the remaining Aroclors must be analyzed every 72 hours for pattern recognition and retention time.

*Note 3 – PEVAL Breakdown Check is not required for PCB-only analyses

Appendix C

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
p,p'-DDT and Endrin breakdown check -required for 8000-series, recommended for 600-series	Initially and every 12 hours	% breakdown of both compounds less than 15%	-re-analyze check solution -perform injector port and/or column maintenance and re-analyze
Initial Calibration- 600-series: 3 point minimum with lowest point at RL 8000-series: 5 point minimum with lowest point at RL	Initially prior to sample analysis, when major instrument maintenance performed, or when CCV falls	**600-series:** 1) RSD of each target <= 10%; OR 2) plot regression curve CC>=0.99 for each target **8000-series:** 1) RSD of each target <= 20%; OR 2) plot regression curve CC>=0.99 for each target	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification (CCV)	After every twenty sample analyses and at the end of the sequence 8000-series: the capping standard must be injected within 12-hours of the PEVAL after ICAL, within 12 hours of the previous CCV 600-series: the capping standard must be injected within 24-hours of the PEVAL after ICAL, within 24-hours of the previous CCV	-Percent difference or drift <= 15% (see SOP for use of "grand mean") -Response of the internal standard must be within a range of +/–50% of the mid-level standard (CCV) in the ICAL	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards

Appendix C

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method blank	Per batch	All targets reported less than RL in LQM	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02
Lab control sample (LCS)	Per batch (If MS/MSD cannot be performed, the LCS must be performed in duplicate)	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02
Matrix spik e(MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02
Internal Standard Response	All samples, method blanks, and QC	-response within a factor of +/–50% of the previous CCV -retention time within window defined by previous CCV	-Evaluate chromatogram and integrations. -Reanalyze or dilute and reanalyze -Flag data
Surrogates	All samples, method blanks, and QC	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02
Dilutions	When extract concentration exceeds calibration range	Report dilution where highest concentration target is in upper half of calibration	-Dilute sample to bring highest concentration into upper half of calibration range. Report all other targets from this dilution. -If lower RL required, prepare dilution 1/10 of dilution that puts highest concentration target into upper half of calibration range.

Appendix C

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Retention time window determination	Annually and with each new column. See guidance in SOP AN66	See guidance in SOP AN66	Use guidance in SOP AN66: *Determination of Retention Time Windows in Gas Chromatographic Analyses*
Demonstration of Capability	Initially and then annually per work group or analyst	Within the 600- or 8000-series method limits (see SOP CA92 and LQM)	-Reanalyze QC sample for the targets that failed to meet the criteria (see SOP CA92)
Method detection limit (MDL)	Annually See SOP CA90	Evaluate data using criteria SOP CA90	-Evaluate data. Check calculations. -Reanalyze MDL samples.

SEVERN TRENT STL

SOP SUMMARY FORM

SOP#: SG45:03.17.04:8	SOP Description: ORGANOCHLORINE PESTICIDES AND PCBs BY GC

Revisions:

___Minor _X_Significant ___Complete Re-write ___New SOP

Summary of Revision(s):

- Revised format to be consistent with current STL Savannah SOP format and NELAC requirements
- Revised safety information to be consistent with current STL Savannah format
- Updated Materials and Apparatus listing
- Added Quality Control, Method Performance and Waste Management information
- Removed Appendix A, retention time table
- Combined 8000-series and 600-series QC information into Appendix B
- Added cap to Grand Mean criteria per SOP AN67
- Added ICV requirement per SOP AN67
- Added requirement that alternate surrogate to DCB must be evaluated if AR1268 is detected
- Removed total area quantitation option for PCBs and Toxaphene, no longer performed
- Removed option NOT to perform capping CCV for internal standard analyses. Although method 8000 allows this option, Method 8081/8082 explicitly states that capping CCV must be performed.
- Removed reference to Separatory Funnel Extractions, no longer performed
- Removed reference to Permanganate clean-ups, no longer performed

IDOCs Required:	___Yes	_X_ No	__NA
MDLs Required:	___Yes	_X_ No	__NA

SOP Implementation Date: 04.17.04

Target Training Completion Date: 04.17.04

Approved by: Andrea Sal

Title: Quality Assurance Manager

Date: 03/22/04

Division Approval:

Title: Laboratory Director

Date: 3/22/04

ELEMENTS BY ICP (200.7 and 6010B)

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Approved by:

R. Wayne Potts — 21 March 2001
Date

Title: Technical Manager, QA

STL X Savannah __ Tallahassee __ Mobile __ Tampa West

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures to determine the concentration of various elements by inductively coupled plasma (ICP) atomic emission spectroscopy. This method contains the analytical procedures for the determination of metals in surface and ground water, wastewater, soil, sediment, leachate (EP or TCLP), and waste samples after digestion.

1.2 Table 1 lists the elements that may be determined by ICP and the characteristic wavelength used for each element. The reporting limit (RL) for each element, the method detection limit (MDL) for each element, and the accuracy and precision criteria for each element are in the Laboratory Quality Manual (LQM) prepared by and for STL Savannah, STL Tallahassee, STL Mobile, and STL Tampa West.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 Prior to analysis by ICP, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and exited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT). The PMTs, located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

2.2 Definitions

ICP -inductively coupled (argon) plasma; sometimes referred to a "ICAP"
TCLP-toxicity compound leaching procedure
EP (tox)-extraction procedure (toxicity)
Analytical Spike or Post-Digestion Spike - addition of a known concentration of analyte to an aliquot of sample after the preparation steps have been performed
RL - reporting limit, the lowest calibration standard or the sample equivalent of the lowest calibration standard; published in LQM or project-specific quality assurance plan (QAPP); sometimes referred to as the "practical quantitation limit(PQL).
MDL - method detection limit, the concentration that can be reported with 99% confidence that the result is greater than zero; published in LQM

2.3 This method is based on EPA Method 200.7 and SW-846 Method 6010B. Note that EPA has promulgated two versions of method 200.7-one for NPDES samples and one for drinking water. The calibration sequence for drinking water by 200.7 requires a multi-point curve with a minimum of three standards and a calibration blank.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 Each digestion lab must have acid spill kits. These kits must be located in a highly accessible area of the lab. Each digestion lab must be equipped with a properly working shower.

3.3 The standards and reagents used to prepare the standards in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.4 The Material Safety Data Sheets (MSDS) for each reagent and standard are located in each laboratory. These sheets denote the type of hazard that each reagent poses, the safe handling instructions for these compounds, and first aid instructions.

4.0 INTERFERENCES

4.1 Spectral interferences are caused by (1) the overlap of a spectral line from another element, (2) unresolved overlap of molecular band spectra, (3) background contribution from continuous phenomena, and (4) stray light from the line emissions of highly concentrated elements.

4.1.1 Spectral overlap may be compensated for by the use of inter-element correction factors.

4.1.2 Background contribution and stray light can be compensated for by a background correction adjacent to the analyte line.

4.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample digestate, by using a peristaltic pump, or by using the method of standards additions(MSA), or use of an internal standard

4.3 Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

5.1 Aqueous Samples

5.1.1 Liquid samples are routinely collected in 250-mL or 500-mL plastic containers. The sample is preserved with HNO_3 to a pH <2. The sample must be digested and analyzed within 6 months of collection. Samples may be stored at room temperature.

5.1.2 Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

5.2 Soil/Sediment Samples
Soil and sediment samples are routinely collected in 500-mL plastic containers. The sample is iced at the time of collection and is stored in the lab at 4C (less than 6C but not frozen) until time of digestion and analysis. The sample must be digested and analyzed within 6 months of collection.

5.3 TCLP or EP Toxicity Leachate Samples
The leachate is transferred to a plastic container after the extraction procedure. The sample is preserved with HNO_3 to a pH <2. The leachate sample must be digested and analyzed within 6 months of completion of the leaching procedure.

5.4 Waste Samples
Waste samples are routinely collected in 500-mL plastic containers. The sample must be digested and analyzed within 6 months of collection.

6.0 APPARATUS AND MATERIALS

6.1 Thermo Jarrell Ash TJA ICAP61E-trace, or other suitable inductively coupled plasma emission spectrometer with data system

6.2 Argon gas supply and appropriate fittings

6.3 Cooling water supply

6.4 Peristaltic pump

6.5 Volumetric flasks

6.6 Pipettes

7.0 REAGENTS

Reagents are tracked in accordance with STL SOP AN44:*Reagent Traceability.*

7.1 Reagent water-lab generated deionized water, ASTM Type I or Type II. The conductivity is monitored in accordance with STL SOP AN35.

7.2 Nitric acid (HNO_3)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals.

7.4 Hydrochloric acid (HCl)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals.

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors. Certificates of analysis or purity must be received with all neat compounds or stock solutions. All preparation steps must be in accordance with STL SOP AN41: *Standard Materials Traceability.* SOP AN43 contains guidance for the preparation of standards.

8.1 Recommended concentrations for the calibration standards are given in Table 1. Appendix A contains examples for the preparation of the initial calibration and calibration verification standards for both 6010 and 200.7. if the laboratory uses "recipes' other than those listed in Appendix A, the recipe must be documented in the standard material traceability logbook or as controlled posting. All standards must have be prepared in 5% hydrochloric acid and 1% nitric acid by volume.

NOTE: Standards must be prepared every six months "or sooner if needed or required." "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

8.2 Preparation of the Linearity Check Solutions

The linearity check solutions are prepared individually according to the following equation:

$$Vs = \frac{Vlc \otimes Clc}{Cs}$$

where
Vs = volume of stock standard (mL)
Cs = concentration of stock standard (mg/L)
Vlc = volume of linearity check standard to prepare (mL)
Clc = concentration of linearity check standard to prepare (mg/L).

The linearity check solutions are prepared at the concentrations specified in Table 1. Prepare sufficient volume to perform the linearity check, maintaining the hydrochloric acid concentration at 5% by volume and the nitric acid concentration at 1% by volume.

9.0 SAMPLE PREPARATION

The sample preparation and digestion procedures are listed in the following SOPs:

MATRIX	SOP
Aqueous and leachate samples	ME50
Soils and Sediments	ME51
Wastes and oils	ME51

10.0 ANALYSIS PROCEDURE

The analytical sequence, including standardization and calibration verification, is included in the SOP Summary in Appendix A. The SOP Summary also included the acceptance criteria for QC, including recommended corrective actions.

10.1 Initial Calibration/Standardization

10.1.1 Turn the ICP on and allow it to become thermally stable before beginning to analyze the calibration standards. It will take about an hour for the instrument to warm up. If optics were turned off, allow 2 hours warm up time.

10.1.2 Run the "Automatic Profile" program. The "automatic profile" of the instrument should be checked twice a day to compensate for changes in air pressure, humidity, and temperature. If the environment of the instrument is such that daily changes in the instrument profile are extreme, the instrument should be "profiled" every few hours.

10.1.3 Analyze the calibration standards and calibrate the ICP. ***If using a multi-point calibration, use the Calibration/Analysis and Curvefit programs to calibrate the instrument.***

10.1.4 The highest concentration calibration standard is reanalyzed after the instrument is standardized as an "unknown". The results for the re-analysis of the highest concentration calibration standard must be within +/- 5% of the true value for each target analyte. If the result for any target analyte is outside of this range, the ICP may need to be "profiled" and the standardization/calibration repeated.

10.1.5 The QC Check standards (ICV) and the Calibration Blank (ICB) are analyzed as a check on the instrument calibration.

10.1.5.1 (EPA Method 6010) The results for the target compounds in the initial calibration verification (ICV) must be within the +/- 10 % of the true value.

10.1.5.2 (EPA Method 200.7) The results for the target compounds in the initial calibration verification (ICV) must be within the +/-5.0 % of the true value. **When performing 200.7 work, note that this solution should be prepared fresh weekly.**

10.1.5.3 (EPA 6010/200.7) The results for the target compounds in the initial calibration blank (ICB) must be less than the RL.

10.1.6 The RL/PQL Check Solution is analyzed to demonstrate that the ICP is capable of detecting the target compounds at or near the reporting limit (RL). The determined concentration must within +/- 50% of the true concentration.

10.1.7 The ICP Interference Check Sample is analyzed. The concentrations of the target analytes must be within 20% of the true concentrations. Pay particular attention to false positives and false negatives for elements not present in the interference check solutions.

10.2 Continuing Calibration Verification (CCV)

10.2.1 The calibration of the ICP must be verified every 10 samples by the analysis of the analysis of the QC Check Solutions (CCV) and the Calibration Blank (CCB).

10.2.1.2 (EPA Method 6010/200.7-DW) The results for the target compounds in the continuing calibration verification (CCV) must be within the +/- 10 % of the true value.

10.2.1.2 (EPA Method 200.7-NPDES) The results for the target compounds in the continuing calibration verification (CCV) must be within the +/-5.0 % of the true value.

10.2.1.3 (EPA 6010/200.7) The results for the target compounds in the continuing calibration blank (CCB) must be less than the Reporting Limit (RL).

10.2.2 ICP Interference Check Solution and the RL check solution are analyzed at the beginning and end of each analytical sequence.

10.3 Sample Analysis

10.3.1 The samples are analyzed only after the ICB/CCB and ICV/CCV criteria are met.

10.3.2 The samples are analyzed in a sequence as follows:

INSTRUMENT WARM-UP
PROFILE
INITIAL CALIBRATION (STANDARDIZATION/CALIBRATION OF THE ICP)
REANALYSIS OF HIGH CONCENTRATION CALIBRATION STANDARD AS A SAMPLE
INITIAL CALIBRATION VERIFICATION (ICV)
INITIAL CALIBRATION BLANK (ICB)
DETECTION LIMIT CHECK SOLUTION
ICP INTERFERENCE CHECK SOLUTION A (ICSA)
ICP INTERFERENCE CHECK SOLUTION AB (ICSAB)
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB

The analytical sequence must end with the analysis of the detection limit check standard, ICSA, ICSAB, CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

10.3.3 Determine the concentration of the samples and QC items using the procedures of Section 11.

10.3.3.1 If the concentration of a sample is above the linear range of the ICP, the sample digestate must be diluted and reanalyzed.

10.3.3.2 The amount of sample digestate needed to prepare the desired dilution is determined form the following equation:

$$V_{digest} = \frac{Vf_{vol}}{DF}$$

where
Vf_{vol} = final volume of diluted sample (mL)
V_{digest} = volume of sample digestate used to make the dilution (mL)

10.3.3.3 The dilution factor is calculated as follows:

$$DF = \frac{Vf_{vol}}{V_{digest}}$$

where
Vf_{vol} = final volume of diluted sample extract (mL)
V_{digest} = volume of sample extract used to make the dilution (mL)

NOTE: The following examples are based on a final volume of 100mL. It may be more convenient to prepare dilutions at smaller final volumes.

EXAMPLE
A sample digestate is analyzed and one of the target analytes exceeds the linear range of the ICP. 1.0mL of the digestate is added to a 100mL volumetric flask and the extract brought up to volume with reagent water. What is the dilution factor?

$$DF = \frac{100mL}{1.0mL} = 100$$

Dilutions must be prepared in reagent water containing 5% hydrochloric acid and 1% nitric acid by volume.

Some samples may require multiple dilutions; that is, a dilution of a dilution will have to be made. In this case, the final dilution factor is the product of the individual dilutions.

10.4 Dilution QC Check
A dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.

10.4.1 Select a sample digestate that contains one or more target analytes at a concentrations greater than 10X the reporting limit.

10.4.2 Dilute the digestate by a factor of 5 (DF=5) and analyze the dilution using the same procedures used for the un-diluted aliquot.

10.4.3 Compare the results of the diluted and un-diluted aliquots of sample digestate.

10.4.4 If the results of the dilution are within ± 10% of the results of the undiluted sample, no matrix interference is present. If the results differ by greater than ± 10%, a matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike (see section 10.4).

If the concentration of the analyte in the sample is not at least 50 times the instrument detection limit, evaluate the post-digestion spike.

10.5 Post-digestion Spike QC Check
A post-digestion spike is performed on one sample per analytical batch to determine if matrix interferences are present. This post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration is not at least 50 times the instrument detection limit. This should be the same sample selected for dilution in 10.3, above.

10.5.1 Transfer 10mL of a digestate to a suitable vial.

10.5.2 Spike the sample with 0.10mL of ICP Matrix Spike I and 0.10mL of ICP Matrix Spike II. The theoretical concentration of the post digestion spike is the same as the LCS or MS if the volume of spiking solution is discounted.

10.5.3 Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed).

10.5.4 Calculate the percent recovery of the post digestion spike:

$$\%REC = \frac{C_{ps} - C_s}{C_2} \times 100$$

where

Cps = concentration of post digestion spike (ug/L)
Cs = concentration of un-spiked sample (ug/L)
C2 = theoretical concentration of spike (ug/L)
(See 10.2.5.2)

10.5.5 Evaluate the recovery using the following decision matrix. Limits for post digestion spikes are 75-125% recovery.

Result of Post Digestion Spikes	Action
Within 75-125% limits	None
>125% recovery	Repeat analysis. Remake spiking solutions, re-spike, and reanalyze. Reanalyze un-spiked sample
<75% recovery but >50% recovery	1) Dilute and re-spike. Elevate RL accordingly (for all associated samples). 2) Spike and evaluate all associated samples. 3) Spike and evaluate all associated samples by single point MSA 4) Qualify all associated samples
<50% recovery	Dilute digestate and repeat spike. Treat all samples associated with spike in the same manner as the spiked sample (i.e., spike or dilute samples) If recoveries are not 75-125%, analyze all associated samples by single point MSA. Note – high level of target analytes may inhibit spike recovery. Consult the supervisor in events where high levels of targets appear to be interfering

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high if corrected for spike recovery.

10.5.6 The post digestion spike and the method of standard additions must not be applied to samples analyzed at a dilution that produces a significant negative response. The analyst must use good judgement when evaluating data where the sample response is negative. Where a significant negative response is present, the digestate should be diluted and reanalyzed to determine the extent of the matrix interferences.

10.6 Single Point Method of Standard Additions

Two identical aliquots of the sample digest, Vx, are taken. One aliquot is spiked with a solution of known concentration, Cs. The second aliquot is analyzed un-spiked (the small volume of standard added to the spiked sample should be disregarded). The concentration of both aliquots are measured and the sample concentration, Cx, is calculated:

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

where

S_1 = absorbance or concentration of the spiked aliquot
S_2 = absorbance or concentration of the un-spiked aliquot
Vs = Volume of spike solution

Example:		
	Sample concentration (S_2):	523 ug/L.
	Spike solution concentration (C_s):	50,000 ug/L
	Volume of spike solution (V_s):	0.10mL
	Volume of sample aliquots (V_x):	10mL
	Spiked sample concentration (S_1):	951 ug/L

C_x = [(523)*(0.10)*(50,000)]/[(951-523)*10)] = [2,615,000]/[4280] = 611 ug/L

10.7 Determination of Linear Range of the ICP

The linear range must be determined a minimum of once per year. Divisions performing CLP analyses are required to determine and document the linear range quarterly. Documentation of the linear range study must be kept on hand and be available for inspection. A summary of the linear range study must be available to the bench analyst.

10.7.1 Profile and calibrate the ICP as described in Section 10.1

10.7.2 Prepare individual standards at concentrations that are expected to define the linear range of the instrument. Use the concentrations in Table 1 for guidance. The calibration standards and the linear range standards should be matrix matched; that is, they have the same percentage of hydrochloric and nitric acids.

10.7.3 Analyze the standards following the analytical sequence described in Section 10.3. Verify the calibration after every 10 analyses.

10.7.4 Compare the concentration of the linear range standard with its true concentration.

$$PercentDifference = \left| \frac{Ccal - Ctrue}{Ctrue} \right| \otimes 100$$

where
Ccal = concentration determined from analysis
Ctrue = true concentration of the standard

If the percent difference is less than or equal to 5%, the linear range is confirmed at that concentration. If the percent difference is greater than 5%, repeat the analysis with a lower concentration.

The linear range may be extended by analyzing higher standards and evaluating the results against the 5% difference criterion. The linear range of the ICP for an analyte is the highest standard of that analyte that meets this criterion.

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Aqueous and Leachate Samples

Aqueous samples are routinely reported in mg/L while the ICP is routinely calibrated in ug/L. If the results are reported in ug/L, the conversion factor is omitted from the calculation.

11.1.1 The concentration of the target analyte in liquid samples is calculated as follows:

$$Concentration\,(mg/L) = ug/L(from\ printout) \otimes \frac{F}{V} \otimes DF \otimes \frac{1mg}{1000ug}$$

where
F = final volume of the sample digestate (L)-usually 50mL (0.050L)
V = volume of sample digested (L)
DF = dilution factor

11.1.2 The Reporting Limit (RL) of the target analyte in liquid samples is calculated as follows:

$$Concentration(mg/L) = RLqap \otimes \frac{F}{V} \otimes DF \otimes \frac{1mg}{1000ug}$$

where
RLqap = reporting limit from STL LQM (ug/L)
F = final volume of the sample digestate (L)
V= volume of sample digested (L)
DF = dilution factor

The LQM Reporting Limits assumes:
F = 50mL, V = 50mL, and DF = 1

11.2 Soil/Solid Samples

Soils and solids are routinely reported in mg/kg while the ICP is routinely calibrated in ug/L. If the results are reported in ug/kg, the conversion factor is omitted from the calculation.

11.2.1 The concentration of the target analyte in soil and solid samples is calculated as follows:

$$Concentration(mg/kg,dw) = ug/L(from\ printout) \otimes \frac{F}{W \otimes solids} \otimes DF \otimes \frac{1mg}{1000ug}$$

where
F = final volume of the sample digestate (L)
W = volume of sample digested (kg)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)
(for example, if the percent solids is 85%, the decimal equivalent is 0.85; if the %solids is 100%, the decimal equivalent is 1.0.)

11.2.2 The Reporting Limit (RL) of the target analyte in soil/solid samples is calculated as follows:

$$Concentration(mg/kg,dw) = RL_{qap} \otimes \frac{0.0010kg}{W \otimes solids} \otimes \frac{F}{0.100L} x\ DF$$

where
RL(qap) = reporting limit from LQM
W = weight of sample digested (kg)
F = final volume of the sample digestate (L)
V = volume of sample digested (L)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)

The LQM Reporting Limits assumes F = 0.100L (100mL), DF = 1, W = 0.0010kg (1.0g), and solids = 1.0

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 STL SOP AN02: *Analytical Batching* and the SOP Summary provide guidance on evaluating QC and sample data, including recommended corrective actions.

12.2 The method detection limit (MDL) is determined annually in accordance with STL SOP CA90. The concentrations of the IDL and MDL solutions are given in Section 8 of this SOP.

12.3 Determination of the Instrument Detection Limit (IDL)

The difference between the MDL and the IDL is the *digestion step*. The MDL samples are prepared and digested prior to analysis. The IDL is defined as three times the standard deviation of seven replicate analyses analyzed over three non-consecutive days. The concentrations of the IDL and MDL solutions are given in Section 8 of this SOP. See SOP CA91 for the procedures for the determination of the IDL.

12.4 The linear range of the ICP must be determined at least annually. The procedure for the determination is given in Section 10.7 of this SOP. If any calibration regression fit, other than linear, is utilized for the calibration of the ICP (i.e., Curvilinear or Full Fit), the upper limit of the linear range is the concentration of the High Standard.

12.5 Interelement correction factors (IEC) for all elements must be determined annually. Use the manufacturer's guidance for determination of the IECs. The IECs must be verified at the beginning and end of each analytical sequence.

13.0 TROUBLESHOOTING AND PREVENTIVE MAINTENANCE

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
ICAP								
Pump Tubing	X							Change.
Nebulizer							X	Clean.
Filters			X					Inspect monthly, clean or replace as needed.
Spray Chamber							X	Clean.
Quartz Torch							X	Clean and realign.

D = daily W = Weekly M = monthly Q = Quarterly SA = semi-annually A = annually AN = as needed

14.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

Excess samples, reagents, and digests must be disposed of in accordance with SOP CA70:*Waste Management*.

15.0 REFERENCES

15.1 *Methods for Chemical Analysis of Water and Waste*; U.S EPA Office of Research and Development: Cincinnati, OHIO, March 1983.

15.2 *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986.

15.3 *Methods for the Determination of Metals in Environmental Samples*; US EPA Office of Research and Development. Washington, DC.

TABLE 1

Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Std. Conc. (mg/L)	Linear Range Std. Conc. (mg/L)*	MATRIX SPIKE CONC. (mg/L)	
						Water (mg/L)	Soil (mg/kg)
Aluminum (Al)	308.215	10	1.0/5.0	0.20	800	2.0	200
Antimony (Sb)	206.838	10	1.0/0.50	0.020	10	0.50	50
Arsenic (As)	189.042 193.696	1.0	1.0/0.50	0.010	25	2.0	200
Barium (Ba)	493.409	10	1.0/5.0	0.010	10	2.0	200
Beryllium (Be)	313.042	1.0	1.0/0.50	0.0040	10	0.050	5.0
Boron (B)	249.678	10	1.0/5.0	0.050	100	1.0	100
Cadmium (Cd)	226.502 228.802	1.0	1.0/5.0	0.0050	10	0.050	5.0
Calcium (Ca)	317.933 315.887	10	1.0/5.0	0.50	800	5.0	500
Chromium (Cr)	267.716	10	1.0/5.0	0.010	25	0.20	20
Cobalt (Co)	228.616	1.0	1.0/0.50	0.010	25	0.50	50
Copper (Cu)	324.754	10	1.0/5.0	0.020	50	0.25	25
Iron (Fe)	259.940 271.441	10	1.0/5.0	0.050	800	1.0	100
Lead (Pb)	220.353	1.0	1.0/0.50	0.0050	5	0.50	50
Magnesium (Mg)	279.079	10	1.0/5.0	0.50	1000	5.0	500

TABLE 1

Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Std. Conc. (mg/L)	Linear Range Std. Conc. (mg/L)*	MATRIX SPIKE CONC. (mg/L)	
						Water (mg/L)	Soil (mg/kg)
Manganese (Mn)	257.610	10	1.0/5.0	0.010	50	0.50	50
Molybdenum (Mo)	202.030	1.0	1.0/0.50	0.010	50	0.50	50
Nickel (Ni)	231.604	5.0	1.0/2.5	0.040	10	0.50	50
Potassium (K)	766.491	20	10/5.0	1.0	50	5.0	500
Selenium (Se)	196.026	10	1.0/5.0	0.010	25	2.0	200
Silver (Ag)	328.068	1.0	1.0/5.0	0.010	5.0	0.050	5.0
Sodium (Na)	588.995 330.231	10	1.0/5.0	0.50	20	5.0	500
Strontium (Sr)	421.552	10	1.0/5.0	0.010	100	0.50	50
Thallium (Tl)	189.042 190.801 377.572	10	1.0/5.0	0.010	30	2.0	200
Tin (Sn)	189.989	10	1.0/5.0	0.050	50	1.0	100
Titanium (W)	334.941	10	1.0/5.0	0.010	10	1.0	100
Vanadium (V)	292.402	10	1.0/5.0	0.010	50	0.50	50
Zinc (Zn)	213.856 206.200+	5.0	1.0/2.5	0.020	20	0.50	50

*For guidance only-instrument sensitivity will vary.

APPENDIX A
SOP SUMMARY

METHOD SUMMARY - ICP ANALYSIS

HOLD/STORAGE

Container	Minimum 250mL plastic bottle with plastic or Teflon-lined lid
Preservative	HNO3 to pH <2 in the field. If dissolved metals are required, filter the samples before preservation.
Storage	Liquids preserved to pH <2 may be stored at room temperature until preparation. Solid samples must be stored at 4C (less than 6C but not frozen) until preparation.
Hold Time	Samples must be analyzed within six months from the time of collection.

SAMPLE PREPARATION

Samples should be prepared with the appropriate matrix-specific procedure.

ANALYTICAL SEQUENCE

Ignite Plasma	Follow instrument manufacturer's guidelines and allow instrument to stabilize for at least 60 minutes.
Profile Instrument	Follow manufacturer's guidelines.
Initial Calibration	Calibrate with a blank and a high standard or a blank and three standards. Verify calibration by reanalyzing highest concentration standard for each element.
Initial Calibration Verification (ICV/ICB)	Analyze an initial calibration verification solution at the beginning of the run. ICV solution must come from a source other than the calibration standard source. Analyze a calibration blank after the ICV.
Continuing Calibration Verification (CCV/CCB)	Analyze a standard with concentrations at or near mid-range levels of the calibration. The CCV should be analyzed every 10 samples and at the end of the analysis run Analyze a continuing calibration blank after every CCV.
Interference Check Solutions	At the beginning and the end of an analysis run, verify the inter-element and background corrections by analyzing the interferent check solutions (ICSA & ICSAB).
Detection limit check solution	At the beginning and the end of an analysis run and verify the accuracy at the RL by analyzing a solution at the SL RL
Serial Dilution	Perform serial dilution (1/5) on a representative sample from each batch..
Post Digestion Spike Recovery.	To check for possible matrix interference, analyze a post digestion spike on a representative sample (minimum of 1 per batch). The post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration in the sample is not at least 50 times the instrument detection limit.

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 std. and 1 blank	
Initial Calibration: Multi-point-minimum 3 stds and 1 blank	Daily	Correlation ≥0.995	Recalibrate
Highest Standard	Immediately after every calibration	Recoveries within ± 5% of expected values	New initial calibration
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	SW846 = within ±10% 200.7 = within ± 5%	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within ±10% of the true value, 200.7-NPDES - within ± 5% 200.7-Drinking Water – within ±10%	Terminate the analysis, fix the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less that the RL/CRDL	Terminate the analysis, correct the problem and reanalyze the previous 10 samples
Interference check standards (ICSA/ICSAB)	At the beginning and end of an analysis run	Determined values must be within ± 20% of the true values. Pay attention to false positives and false negatives for elements not present in the solutions.	Terminate the analysis, correct the problem, recalibrate, and reanalyze all samples since the last ICS that was in control.
Lab control sample	One per batch of twenty samples or less	6010B: STL LQM 200.7: 85-115%	Redigest and reanalyze batch
Preparation blank - SW846	One per batch of twenty samples or less	|result| <RL or result <5% of the analyte level in the sample.	Redigest and reanalyze batch
Preparation blank - 200.7	One per batch of twenty samples or less	|result| <RL or result <10% of the analyte level in the sample	Redigest and reanalyze batch
MS/MSD - SW846	One set per batch of twenty samples or less	STL LQM	Flag and report data
Serial Dilution (1/5 Dilution)	One per batch of twenty samples or less	See section 10.3.4	
Post Digestion Spike	One per batch of twenty samples or less	See section 10.4.5	
Detection Limit Check Solution	At the beginning and end of an analysis run	Recovery +/-50% of the true concentration.	Stop the analysis, fix the problem and reanalyze the affected samples.

APPENDIX B
EXAMPLES OF STANDARD PREPARATION

GENERAL INSTRUCTIONS

All calibration standards must contain 5% hydrochloric acid and 1% nitric acid by volume. The following table lists the volume of each acid needed to prepare the desired final volume of standard.

Final Volume of Standard (mL)	Volume of Hydrochloric acid (mL)	Volume of Nitric Acid (mL)
100	5.0	1.0
200	10	2.0
500	25	5.0
1000	50	10

For example, to prepare 500mL of a standard:

- Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask.
- Add 5.0mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask.
- Add the volumes of the stock standards given in the table to the volumetric flask:
- Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature.

SINGLE POINT CALIBRATION STANDARDS FOR 6010

Calibration Standard 1-Calibration Blank (ICB, CCB)

Add 500mL to 600mL of reagent water to a clean 1-L volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. Dilute to a final volume of 1.0-L with reagent water. Store the standard at room temperature. Other volumes may be prepared at the discretion of the lab. The nitric acid concentration must be 1% by volume and the hydrochloric acid concentration must be 5% by volume.

Calibration STANDARD 2

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (ml)	Conc. of Cal Std (mg/L)
Silver(Ag)	1000	0.50	500	1.0
Arsenic(As)	1000	0.50		1.0
Molybdenum(Mo)	1000	0.50		1.0
Lead(Pb)	1000	0.50		1.0
Selenium(Se)	1000	5.0		10
Antimony(Sb)	1000	0.50		1.0
Thallium(Tl)	1000	5.0		10

Calibration STANDARD 3

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Beryllium(Be)	1000	0.50	500	1.0
Barium(Ba)	1000	5.0		10
Cadmium(Cd)	1000	0.50		1.0
Cobalt(Co)	1000	0.50		1.0
Chromium(Cr)	1000	5.0		10
Copper(Cu)	1000	5.0		10
Manganese(Mn)	1000	5.0		10
Nickel(Ni)	1000	2.5		5.0
Zinc(Zn)	1000	2.5		5.0

Calibration STANDARD 4

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Aluminum(Al)	10000	0.50	500	10
Iron(Fe)	10000	0.50		10
Boron(B)	1000	5.0		10
Strontium(Sr)	1000	5.0		10
Titanium (Ti)	1000	5.0		10

Calibration STANDARD 5

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Calcium(Ca)	10000	0.50	500	10
Potassium(K)	10000	1.0		20
Magnesium(Mg)	10000	0.50		10
Sodium(Na)	10000	0.50		10
Tin(Sn)	1000	5.0		10
Vanadium(V)	1000	5.0		10

MULTI-POINT INSTRUMENT CALIBRATION-200.7

For all drinking water samples (EPA 200.7) the ICP must be calibrated with a minimum of three standards and a blank. The following standards may be used for this purpose. With the Thermo Jarrell Ash software the Calibration Analysis and Curve-fit programs must be used to be successful with the multi-point calibration of the ICP instruments.

High Standard.

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Aluminum (Al)	10000	1.0	1000	10
Antimony (Sb)	1000	1.0		1.0
Arsenic (As)	1000	1.0		1.0
Boron (B)	1000	10		10
Barium (Ba)	1000	10		10
Beryllium (Be)	1000	1.0		1.0
Cadmium (Cd)	1000	1.0		1.0
Calcium (Ca)	10000	1.0		10
Cobalt (Co)	1000	1.0		1.0
Chromium (Cr)	1000	10		10
Copper (Cu)	1000	10		10
Iron (Fe)	10000	1.0		10
Lead (Pb)	1000	1.0		10
Magnesium (Mg)	10000	1.0		10
Manganese (Mn)	1000	10		10
Molybdenum Mo)	1000	1.0		1.0
Nickel (Ni)	1000	5.0		5.0
Potassium (K)	10000	1.0		10
Selenium (Se)	1000	10		10
Silver (Ag)	1000	1.0		1.0
Sodium (Na)	10000	1.0		10
Strontium (Sr)	1000	10		10
Thallium (Tl)	1000	10		10
Tin (Sn)	1000	10		10
Titanium (Ti)	1000	10		10
Vanadium (V)	1000	10		10
Zinc (Zn)	1000	5.0		5.0

Mid-Level Standard- Prepare as the CCV is prepared.

Low-Level Standard- Prepare as the RL/PQL Check Standard.

Initial Calibration Verification (ICV) Solution

Element/Stock	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of CCV Std (mg/L)
SPEX QC19	(1)	5.0	500	(2)
SPEX QC 7	(1)	5.0		(2)
Tin(Sn)	1000	0.50		1.0
Strontium (Sr)	1000	0.50		1.0
Potassium (K)	10000	0.50		10(3)
Sodium (Na)	10000	0.45		10(3)

(1) SPEX QC19 and SPEX QC7 are solutions containing multiple elements. The concentrations are given on the certificate of analysis.

(2) The final concentrations of the various elements are the same as listed in Table 1. The SPEC QC solutions are diluted by a factor of 100 from the concentration listed on the certificate of analysis.

(3) These concentrations include the contribution from SPEX Solutions QC-7

Continuing Calibration Verification (CCV) Standard

(also used as midpoint of multi-point calibration for EPA 200.7)

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of CCV Std (mg/L)
Aluminum (Al)	10000	0.50	1000	5.0
Antimony (Sb)	1000	0.50		0.50
Arsenic (As)	1000	0.50		050
Boron (B)	1000	5.0		5.0
Barium (Ba)	1000	5.0		5.0
Beryllium (Be)	1000	0.50		0.50
Cadmium (Cd)	1000	0.50		0.50
Calcium (Ca)	10000	0.50		5.0
Cobalt (Co)	1000	0.50		0.50
Chromium (Cr)	1000	5.0		5.0
Copper (Cu)	1000	5.0		5.0
Iron (Fe)	10000	0.50		5.0
Lead (Pb)	1000	0.50		0.50
Magnesium (Mg)	10000	0.50		5.0
Manganese (Mn)	1000	5.0		5.0
Molybdenum (Mo)	1000	0.50		0.50
Nickel (Ni)	1000	2.5		2.5
Potassium (K)	10000	0.50		5.0
Selenium (Se)	1000	5.0		5.0
Silver (Ag)	1000	0.50		0.50
Sodium (Na)	10000	0.50		5.0
Strontium (Sr)	1000	5.0		5.0
Thallium (Tl)	1000	5.0		5.0
Tin (Sn)	1000	5.0		5.0
Titanium (Ti)	1000	5.0		5.0
Vanadium (V)	1000	5.0		5.0
Zinc (Zn)	1000	2.5		2.5

Reporting Limit (RL) Check Standard

(also used as low point in multi-point calibrations; e.g. EPA 200.7)

Preparation of RL/PQL Stock A-ICP

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. Of Stock Std (mg/L)
Silver (Ag)	1000	0.10	100	1.0
Arsenic (As)	1000	0.10		1.0
Cadmium (Cd)	1000	0.050		0.50
Copper (Cu)	1000	0.20		2.0
Nickel (Ni)	1000	0.40		4.0
Lead (Pb)	1000	0.050		0.50
Selenium (Se)	1000	0.10		1.0
Thallium (Tl)	1000	0.10		1.0

Preparation of RL/PQL Stock B-ICP

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. Of Stock Std (mg/L)
Aluminum (Al)	10000	0.20	100	20
Boron (B)	1000	0.50		5.0
Barium (Ba)	1000	0.10		1.0
Beryllium (Be)	1000	0.040		0.40
Calcium (Ca)	10000	0.50		50
Cobalt (Co)	1000	0.10		1.0
Chromium (Cr)	1000	0.10		1.0
Iron (Fe)	10000	0.050		5.0
Magnesium (Mg)	10000	0.50		50
Manganese (Mn)	1000	0.10		1.0
Molybdenum Mo)	1000	0.10		1.0
Sodium (Na)	10000	0.50		50
Antimony (Sb)	1000	0.20		2.0
Strontium (Sr)	1000	0.10		1.0
Tin (Sn)	1000	0.50		5.0
Titanium (Ti)	1000	0.10		1.0
Vanadium (V)	1000	0.10		1.0
Zinc (Zn)	1000	0.20		2.0

Preparation of RL/PQL Stock C-ICP

Element	Conc. of Stock Std	mL of Stock Std	Final Volume of Cal Std	Conc. Of Stock Std
Potassium(K)	10000	1.0	100	100

Preparation of the RL/PQL Check Solution-ICP

RL/PQL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A-ICP	5.0	500
Stock B-ICP	5.0	
Stock C-ICP	5.0	

ICP Interference Check Solutions

Preparation of ICP Interference Check Solution A

Element	Conc. Of Stock(mg/L)	mL of Stock Std	Final Volume(mL)	Conc. (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25		500
Magnesium (Mg)	10000	25		500
Iron (Fe)	10000	10		200

Preparation of ICP Interference Check Solution AB

Element	Conc. of Stock(mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25		500
Magnesium (Mg)	10000	25		500
Iron (Fe)	10000	10		200
Silver (Ag)	1000	0.10		0.20
Arsenic (As)	1000	0.050		0.10
Barium (Ba)	1000	0.25		0.50
Beryllium (Be)	1000	0.25		0.50
Cadmium (Cd)	1000	0.50		1.0
Cobalt (Co)	1000	0.25		0.50
Chromium (Cr)	1000	0.25		0.50
Copper (Cu)	1000	0.25		0.50
Manganese (Mn)	1000	0.25		0.50
Nickel (Ni)	1000	0.50		1.0
Lead (Pb)	1000	0.025		0.050
Antimony (Sb)	1000	0.30		0.60
Selenium (Se)	1000	0.025		0.050
Thallium (Tl)	1000	0.050		0.10
Vanadium (V)	1000	0.25		0.50
Zinc (Zn)	1000	0.50		1.0
Molybdenum (Mo)	1000	0.50		1.0
Tin (Sn)	1000	0.50		1.0

ICP Matrix Spiking Solutions

ICP Matrix Spiking Solution 1 is a solution purchased from SPEX. The certificate of analysis will list the concentrations of the analytes. Store this solution at room temperature. Prepare this solution every six months or sooner if needed or required.

Preparation of the ICP Matrix Spiking Solution 2

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Boron (B)	1000	10	100	100
Calcium (Ca)	10000	5.0		500
Magnesium (Mg)	10000	5.0		500
Molybdenum (Mo)	1000	5.0		50
Potassium (K)	10000	5.0		500
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	5.0		50
Tin (Sn)	1000	10		100
Titanium (Ti)	1000	10		100

IDL/MDL Solution

The IDL/MDL solution is used in this procedure for two purposes:

1) To determine the Instrument Detection Limit (IDL) of each target compound on a quarterly basis (SOP CA91); and
2) To determine the Method Detection Limit (MDL) of each target compound on an annual basis (SOP CA90). MDLs should be digested straight and at 1:2 dilutions.

Preparation of IDL/MDL Stock A

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Silver (Ag)	1000	0.040	100	0.40
Arsenic (As)	1000	0.20		2.0
Barium (Ba)	1000	0.020		0.20
Beryllium (Be)	100	0.050		0.050
Cadmium (Cd)	1000	0.040		0.40
Lead (Pb)	1000	0.10		1.0
Antimony (Sb)	1000	0.20		2.0
Selenium (Se)	1000	0.20		2.0
Thallium (Tl)	1000	0.20		2.0

Preparation of IDL/MDL Stock B

Element	Conc. Of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. Of Std. (mg/L)
Cobalt (Co)	1000	0.030	100	0.30
Chromium (Cr)	1000	0.10		1.0
Copper (Cu)	1000	0.10		1.0
Manganese (Mn)	1000	0.020		0.20
Molybdenum (Mo)	1000	0.040		0.40
Nickel (Ni)	1000	0.10		1.0
Tin (Sn)	1000	0.20		2.0
Vanadium (V)	1000	0.060		0.60
Zinc (Zn)	1000	0.10		1.0

Preparation of IDL/MDL Stock C

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Aluminum (Al)	10000	0.10	100	10
Calcium (Ca)	10000	0.10		10
Iron (Fe)	10000	0.10		10
Magnesium (Mg)	10000	0.10		10
Potassium (K)	10000	0.20		20
Sodium (Na)	10000	0.040		4.0

IDL/MDL Solution

Preparation of IDL/MDL Stock D

Element	Conc. Of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Boron(B)	1000	0.40	100	4.0
Strontium (Sr)	1000	0.040		0.40
Titanium (Ti)	1000	0.050		0.50
Sodium (Na)	10000	2.0		200

Preparation of the IDL/MDL Check Solution

IDL/MDL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A	5.0	1000
Stock B	5.0	
Stock C	5.0	
Stock D	5.0	

The IDL/MDL Check Solution contains the following elements at the given concentrations:

Element	Concentration(mg/L)
Be	0.00025
Ba, Mn	0.0010
Co	0.0015
Ag, Cd, Mo, Sr	0.0020
Ti	0.0025
V	0.0030
Cr, Cu, Ni, Pb, Zn	0.0050
As, Sb, Se, Sn, Tl	0.010
Na(1), B	0.020
Ca, Fe, Mg	0.050
Al, K	0.10
Na	1.0

(1) If the wavelength for sodium is 588.995, the lower concentration (0.020mg/L) is used for the IDL./MDL check solution. In this case, only stocks A,B, and C are used to make the IDL/MDL Check Solution. IDL/MDL Check Solutions for B, Sr, and Ti are prepared and evaluated separately.