QUALITY ASSURANCE PROJECT PLAN

FOR

CHEMICAL WASTE MANAGEMENT, INC.
Vickery, Ohio

ADDENDUM TO THE GROUND WATER MONITORING PLAN

MAY 29, 1992

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PREPARED BY:

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QUALITY ASSURANCE PROJECT PLAN
FOR
CHEMICAL WASTE MANAGEMENT, INC
VICKERY, OHIO

ADDENDUM TO THE
GROUND WATER MONITORING PLAN

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III. PROJECT DESCRIPTION

1. INTRODUCTION

The United States Environmental Protection Agency (USEPA) has granted to Chemical Waste Management, Inc. (CWM) a Final Exemption for Continued Injection of Hazardous Waste Subject to the Land Disposal Restriction of the Hazardous and Solid Waste Amendments of 1984. This exemption was issued on August 7, 1990, and became effective upon issuance of CWM's permit modifications by the Ohio Environmental Protection Agency (OEPA) on August 10, 1990. The Exemption applies to the four (4) Class 1 injection wells CWM operates at its Vickery, Ohio commercial waste disposal facility.

Both the USEPA Exemption and the OEPA permit modifications require CWM to meet certain specific conditions enumerated in the documents. One of the specific conditions common to both Agencies is the requirement that CWM submit an approvable ground water monitoring plan (GWMP) for the installation of wells capable of verifying no-migration of injected wastes from the injection zone or into USDWs. The conditions require a monitor well completed in the Trenton Formation or below, capable of monitoring pressure and allowing for fluid sampling. In

addition, the OEPA conditions call for a well capable of monitoring the ground water quality in the lowermost USDW. The USEPA condition additionally requires that the GWMP conform to the specifications set forth in Attachments B and C of a letter from the USEPA to CWM, dated April 18, 1990. These attachments contain specific recommendations for siting and design of the CWM GWMP, and are found in GWMP Appendix C. Additionally, the USEPA has provided CWM with the documents "Region V Guidelines for Class 1 Well Monitoring Plans", and "United States Environmental Protection Agency Region V Supplemental Guidelines For Class I Monitor Well Plans And Operations" to assist in the preparation of the GWMP. Copies of these guidance documents are included in GWMP Appendix D. CWM submitted an initial GWMP to USEPA and OEPA in October 1990, and revised plans in December 1990, May 1991 and August 1991.

CWM has prepared a GWMP which provides for feasible and workable well designs that are simultaneously supportive of the no-migration standards and protective of USDWs. CWM is proposing to complete a deep monitor well in the Knox-Kerbel Formation and a shallower monitor well in the Brassfield Formation. A more detailed description of the project, including the sequence of well construction and the methods of installation are found in Sections 2, 3 and 4 of the

GWMP.

This QAPjP generally follows the outline specified in "CONTENT REQUIREMENTS FOR RCRA FACILITY INVESTIGATION (RFI) QUALITY ASSURANCE PROJECT PLANS (QAPjPs)", from the USEPA Region V Quality Assurance Section, Monitoring and Quality Assurance Branch, Environmental Sciences Division. Modifications are introduced to address the QAPP requirements specified in the Region V GWMP guidelines referenced on the previous page. If any differences in procedures or analytical methods exist between the GWMP document and this QAPjP, the QAPjP procedures or methods take precedence.

2. SITE DESCRIPTION

CWM operates a commercial waste disposal facility that receives aqueous waste for treatment, storage and ultimate disposal by deep well injection. The principle features of the present facility are the waste unloading area (waste is received from tank trucks), truck wash, storage tanks, container storage, surface treatment facilities and four active injection wells.

Waste is injected into the Cambrian age Mt. Simon Sandstone at a depth

below ground surface of approximately 2800 ft to 2900 ft. The injection interval is separated from the base of the lowermost USDW by approximately 2000 ft of nearly flat lying interbedded shales, dolomites, limestones and thin sandstones.

The CWM Vickery facility is located in a rural, unincorporated area of Sandusky County in north-central Ohio. The facility is located in Sections 23 and 26 of the Riley Township, approximately 6 miles east of the town of Fremont and 2 miles west-southwest of the unincorporated community of Vickery. The facility is bordered on the east by State Highway 510, and the west boundary extends to County Road 244 (Karbler Road). The topography of the CWM Vickery site is relatively flat with a slight slope to the north. The site elevation ranges from 609 to 611 ft. Raccoon and Little Raccoon Creeks drain to the north and discharge at Sandusky Bay on Lake Erie, about five miles north of the site. The facility is not located within the hundred year flood plain.

Seven wells have been drilled on the CWM Vickery site for the purpose of injection of treated hazardous waste. Of the seven wells drilled, four are currently in operation and three have been properly plugged and abandoned.

The CWM petition document, to which the GWMP and this QAPjP are subordinate, documented in detail the siting and operation of the Vickery facility. Specific areas exhaustively studied were: artificial penetrations, regional geology, site specific geology, core analysis, waste analysis and reservoir modeling. Additional details of site specific features are found in the Chemical Waste Management Petition for the Vickery facility.

3. SITE HISTORY AND BACKGROUND

A waste handling facility was originally established at the current Vickery site in 1961 by Don's Waste Oil, which was formed in 1958 to recycle used oil from service stations. In 1961 Don's began to accept additional industrial wastes and store them in a pond at Vickery. Additional storage ponds were constructed throughout the late 1960's

In 1971 the company was incorporated as Ohio Liquid Disposal, Inc. (OLD). In 1972 OLD received a state permit, and drilled a test hole (later to become Disposal Well No. 1) to evaluate subsurface conditions for the suitability of injecting the stored wastes. In 1975 a permit to use the well for waste disposal was issued by the state, and injection began in 1976. Six additional wells were drilled and

completed between 1976 and 1981, all designed to inject waste into the Mt. Simon Sandstone. CWM purchased the facility in the early 1980's.

During the late 1970's and early 1980's the wells had experienced several problems with casing/tubing corrosion which resulted in releases of waste liquids into unpermitted upper portions of the injection zone. During the period 1983 - 1985 CWM recompleted all the wells using design and construction methods superior to the original, and installed automated injection control and monitoring equipment.

4. PROJECT OBJECTIVES

4.1 SCOPE OF THE REGULATORY CONCERN

Injection activities at the CWM Vickery facility have been extensively modeled utilizing the SWIFT II simulator (see Petition Section 14). The simulation modeled the vertical and lateral pressurization and waste plume movement for a period of 10,000 years. Modeling indicated the CWM Vickery site was capable of safely containing all injected wastes, and the operations were protective of USDWs. Input to the model was conservative for all factors, consisting of maximum injection rates and pressures permitted over an operational period of twenty years,

maximum mobility factors for injected waste constituents and reservoir petrophysical parameters less supportive of containment than those actually measured at the site.

USEPA and OEPA require additional and ongoing confirmation of the injection zone's ability to properly contain injected waste in the manner predicted by the simulation. The installation of additional wells capable of monitoring formation pressures and chemical composition is intended to accomplish this goal.

4.2 OBJECTIVE OF THE GWMP

The GWMP provides for the design, installation and ongoing operation of two monitoring wells capable of confirming that waste is not migrating out of the injection zone into USDWs, and that pressurization of the subsurface formations is consistent with that predicted by the SWIFT II model, thereby increasing confidence in the no-migration petition. The methods to be utilized for confirmation of the no-migration petition model predictions are: continuous monitoring of reservoir pressure within the lower Knox-upper Kerbel Formations; periodic sampling and analysis of the interstitial fluids from the Knox-Kerbel zone; periodic monitoring of potentiometric head in the Brassfield Formation; periodic sampling

and analysis of interstitial fluids from the Brassfield Formation.

Of the above methods, continuous pressure monitoring in the Knox/Kerbel zone of the deeper monitor well is the most critical in determining the validity of the no-migration model simulation and early detection of any anomalous activity within the injection zone. The pressure front caused by waste injection operations propagates outward and upward through the injection zone formations well in advance of the physical movement of injectate. Anomalous changes in reservoir pressurization can be detected at a much earlier point in time than actual chemical changes caused by injectate movement into the monitored zone. Detection of changes in formation chemistry may also be disadvantaged by possible analytical interferences introduced by high concentrations of naturally occurring chemical constituents in the formation brine, or the presence of contamination from prior injectate releases into the upper injection zone.

4.3 INTENDED DATA USAGE

Data generated during the implementation of the GWMP will be utilized to verify the SWIFT II reservoir simulation performed for the CWM Vickery Petition demonstration. The numerical simulation, performed with very conservative input

parameters, indicated that the injection of waste at the Vickery facility meets the USEPA no-migration standards, does not endanger USDWs, and is protective of human health and the environment.

The baseline for monitor zone formation pressure and interstitial water chemistry will be established over a twenty-four (24) month period after the monitor wells are installed. The schedule of monitoring and sampling each well is detailed in Section 7 of the GWMP. Periodic reports as specified in Section 10 of the GWMP will be submitted to the USEPA and OEPA documenting the conditions within the monitored zones.

4.3.1 Field Measurement Usage

During well purging prior to collection of a fluid sample, produced formation fluid temperature, pH, conductivity and dissolved oxygen will be measured to insure that the fluid parameters have stabilized before taking the sample. This insures that the sample to be analyzed is representative of the formation fluid, and provides a general characterization of the ground water quality.

During down hole testing with the TAM packer tool a measurement of

formation pressure and hydraulic conductivity will be obtained in the Brassfield, Cincinnatian, Trenton and Knox intervals. These measurements will provide background data on the pressure gradient existent at the site and the potential flow characteristics of the tested formations.

Pump tests will be conducted in the completion intervals of the Brassfield and Knox - Kerbel Wells. Hydraulic conductivity and specific yield will be calculated and utilized in estimating practical purging rates to be used in the long term sampling of the wells and the associated resultant drawdowns.

Prior to initiating purging and sampling operations, OVA or HNu measurements will be taken from the well heads of the Knox-Kerbel and Brassfield wells. This will prevent the exposure of field personnel to potentially explosive or toxic environmental hazards.

5. TARGET PARAMETERS

5.1 FORMATION PRESSURE MEASUREMENTS

In the Knox/Kerbel monitor well, formation pressure in the permanent completion zone will be monitored continuously with a 50 psig range pressure

transducer capable of ±0.05 psi accuracy, suspended in the piezometer tubing (see Sections 3, 4, 7 and Appendix O of the GWMP). During the drilling of this well stratigraphically higher zones (Brassfield, Cincinnatian, Trenton, upper Knox) will be tested with the TAM Packer system (see Appendix G of the GWMP). Pressure measurements will be made utilizing a strain gauge with resolution of 0.005% of full scale, and accuracy of 0.1% of full scale (gauge scale selected to be zone specific).

In the Brassfield monitor well, the static water level will be checked and reported at the time of the periodic sampling events (see Section 10 of the GWMP) via the use of an electric measuring tape. Accuracy will be to ±0.1 feet.

5.2 CHEMICAL AND PHYSICAL PARAMETERS

Fluid from the Knox-Kerbel and Brassfield Formation monitor wells will be analyzed for differing suites of parameters. Also, in the Knox-Kerbel well, different parameters will be analyzed for in the samples from the completed deep monitoring zone (Knox-Kerbel) versus the shallower intervals from which samples will be recovered via TAM packer. These parameter lists have been developed to include typical natural ground water constituents as well as parameters that may

have been introduced from injection activities.

5.2.1 Reporting Limits, Sample Dilution

Laboratory reporting limits for analytical data are in tables contained in QAPjP Appendix A-10, along with background data concerning the development of these limits. The reporting limit is analogous to the EPA's definition of a Practical Quantitation Limit (PQL), and is the value above which reliable data can be reported. Additional data on reporting limits and method detection limits can be found in the individual Standard Operating Procedures (SOPs) referenced from Section IX of this QAPjP. The reporting limit for each chemical parameter will also be indicated on the analytical reports provided to CWM and regulatory agencies by the laboratory conducting the analysis.

High concentrations of total dissolved solids are anticipated to be encountered in many of the environmental and drilling mud samples from the Knox-Kerbel and Brassfield wells. This high level of dissolved solids may cause severe interference with some analytical processes, or cause the working ranges of equipment to be exceeded, particularly for inorganic analytes. Dilution of some analytical samples may be necessary to obtain reliable data for certain analytes.

The dilution factor required will be determined by the laboratory analyst based on knowledge of the specific constituents present and the parameter being analyzed. Sample dilution will result in elevated detection and reporting limits. The dilution factor used and the correspondingly higher detection and reporting limits will be indicated on all analytical reports. QAPjP Appendix A-10 (page 4 of 18) further addresses sample dilution.

5.2.2 Knox-Kerbel Monitor Well Analysis Parameters

Fluids recovered from the Brassfield Formation during drilling of the Knox-Kerbel monitor well will be tested only for total dissolved solids, plus the temperature, pH and fluid resistivity which are recorded by the TAM packer down hole test tool as the sample is being collected.

Fluids recovered from the Cincinnatian Series, Trenton Formation and upper Knox Formation, which are obtained with the TAM packer system during drilling, are to be tested for the following physical and chemical parameters, as specified by the USEPA in project specific guidance, and hence-forth referred to as the TAM Packer Parameter List:

Ethylbenzene
1,2-Dichloroethane

Toluene
Chlorobenzene
Tetrachloroethene
1,2-Dichloroethene (total)
trans-1,3-Dichloro-propene
cis-1,3-Dichloropropene
Carbon Tetrachloride
Chloroform
1,1,1-Trichloroethane
Bromomethane
Chloromethane
Chloroethane
Vinyl Chloride
Methylene Chloride
Bromoform
Bromodichloromethane
1,1-Dichloroethane
1,1-Dichloroethene
1,2-Dichloropropane
1,1,2-Trichloroethane
Tricloroethene
1,1,2,2-Tetrachloroethane
Methanol
Methyl Ethyl Ketone
Total Phenols
PCB's
Dissolved Iron
Dissolved Nickel
Dissolved Arsenic
Dissolved Cadmium
Dissolved Chromium
Dissolved Lead
Dissolved Zinc
Dissolved Barium
Dissolved Mercury
Dissolved Selenium
Dissolved Silver
Fluoride

Calcium
Magnesium
Manganese
Potassium
Sodium
Boron
Bromide
Chlorides
Nitrate
Nitrogen Ammonia
Sulfates
Total Dissolved Carbonate
Total Dissolved Solids
Alkalinity
pH
Specific Gravity
Specific Conductance
Temperature

Where analysis for dissolved constituents is specified, samples will be field filtered to .45μm. Samples for inorganic analysis may also be field filtered.

The planned monitoring interval in the Knox-Kerbel Formation (2410-2440 feet) will, upon completion of the well, be subjected to a step rate pump test for the determination of hydraulic parameters and optimum well purging rate. Utilizing methods defined earlier in this GWMP (Section 7), the well will be properly purged, and fluid samples recovered at the surface for initial parameter analysis. Chemical and physical parameters to be analyzed for, as specified by the USEPA in project specific guidance, and hence-forth referred to as the Appendix IX Inclusive Parameter List, include:

Calcium
Magnesium
Manganese
Potassium
Sodium
Boron
Bromide
Chlorides
Fluoride
Nitrate
Nitrogen Ammonia
Sulfate
Total Dissolved Carbonate
Specific Conductance
pH
Total Dissolved Solids
Specific Gravity
Alkalinity
Complete Appendix IX Parameter List (From 40 CFR Part 264)

Appendix IX parameters of 40 CFR Part 264 were presented in GWMP Table 7-1.

Parameters listed in Appendix IX of 40 CFR Part 264 which, in the initial sampling event following well completion, are detected at levels above the Practical Quantitation Limit (as defined in the methods of RCRA Waste Analysis Guidance SW-846, third edition, 1986), will continue to be included on the list of parameters tested for in future Knox-Kerbel monitor zone sampling events (Continuing Parameter List). Laboratory reporting limits which are given in QAPjP Appendix A-10 are analogous to the RCRA PQL definition. Appendix IX parameters not detected above the PQL in the initial sampling will be dropped from the testing schedule.

5.2.3 Brassfield Monitor Well Analysis Parameters

Analyses will be performed for the following chemical or physical parameters for fluids recovered from the Brassfield well monitoring zone:

pH
Specific gravity
Total Organic Carbon (TOC)
Sulfate (SO_4)
Acidity (as $CaCO_3$)
Total Solids @ 105 °C
Total Suspended Solids
Chlorides
Dissolved Chromium
Dissolved Iron
Dissolved Nickel
Dissolved Zinc
Dissolved Lead
Conductivity @ 25 °C

Where analysis for dissolved constituents is specified, samples will be field filtered to .45 μm.

6. SAMPLE NETWORK AND RATIONALE

6.1 SITING AND SAMPLING DEPTHS

CWM has proposed to install two monitor wells at the Vickery, Ohio facility to monitor for no-migration out of the injection zone, and to monitor ground water quality in the lowermost USDW. The deeper monitor well will be drilled and completed in the Knox-Kerbel Formations at ±2410-2440 ft prior to the drilling and

completion of the shallower monitor well, proposed to be completed in the Brassfield Formation at ±700-720 ft. See Figures 2-1 and 2-6 in the GWMP for a graphical presentation of the stratigraphic intervals to be completed, and their relationship to the injection interval and USDW. Both of the intervals to be monitored must meet the requirements of the USEPA Region V monitoring plan guidelines for stratigraphic location, aerial extent, zone thickness and permeability, and were mandated by the USEPA Region V UIC Section.

The Knox-Kerbel Formation monitor well will be located to the north-northeast, and within 100 ft of CWM's existing Disposal Well No. 2. The Brassfield Formation monitor well will be located to the east, and within 100 ft of CWM's existing Disposal Well No. 2. These locations are near the current and historical center of injection at the CWM site and near a presently active injection well. These proposed locations offer sufficient operational space for the safe installation and operation of the wells, avoiding as much as possible the existing infrastructure of the CWM facility, including a high pressure pipeline to the northwest of the No. 2 well. The exact location will be submitted with the final completion report. See Figures 2-1 and 2-4 of the GWMP for a plan view of the approximate location of the proposed monitor wells in relation to the existing injection wells at the facility.

Additional information on siting, including stratigraphic, mineralogical, petrophysical and hydrologic data on the geological formations to be monitored and tested was detailed in Section 2 of the GWMP.

6.2 SAMPLE MATRICES, PARAMETERS, FREQUENCY OF COLLECTION

The sampling and testing to be performed during the installation, completion and ongoing monitoring of the Knox-Kerbel and Brassfield wells was presented in Sections 4, 7, 8, and 10 of the GWMP. This information is summarized here in Table III-1 and Table III-2.

7. PROJECT SCHEDULE

The initiation of installation of the groundwater monitoring wells is dependent upon numerous factors, including approval of a finalized GWMP plan and QAPP by the USEPA and OEPA, selection of contractors and vendors capable of meeting the specifications and requirements in the final plans, equipment and material availability and seasonal road use restrictions on heavy equipment movement imposed by the Ohio Department of Transportation. Once installation commences, the Knox-Kerbel well will be drilled, completed and sampled first, followed closely by the installation of the Brassfield well. The schedules for drilling and completion

are indicated in Figure III-1 and Figure III-2. Reports will be provided as indicated in Section 10 of the GWMP.

TABLE III-1 (Page 1 of 2)
SUMMARY OF SAMPLING AND ANALYSIS
KNOX-KERBEL MONITOR WELL

MATRIX	FIELD PARAMETERS	LAB PARAMETERS	INVESTIGATIVE SAMPLES Total No.	FIELD QUALITY CONTROL SAMPLES DUPLICATE SAMPLES	FIELD BLANKS	TRIP BLANKS	MATRIX TOTALS
Make-up Water (1)	Temp, pH, Cond, DO	TAM PACKER LIST					
		Volatile Organics	1	1	1	1	4
		Extractables	1	1	1	--	3
		Metals / Inorganic ions (Field filtered)	1	1	1	--	3
		PCB's	1	1	1	--	3
		Physical chemistry	1	1	1	--	3
Drilling Mud (2)	Temp, pH, Cond, DO	TAM PACKER LIST					
		Volatile Organics	3	3	3	3	12
		Extractables	3	3	3	--	9
		Metals / Inorganic ions (Field filtered)	3	3	3	--	9
		PCB's	3	3	3	--	9
		Physical chemistry	3	3	3	--	9
Ground Water (3)	Temp, pH, Cond, DO Pressure, Hydraulics	TDS ONLY					
		Total dissolved solids	1	1	1	--	3
Ground Water (4)	Temp, pH, Cond, DO Pressure, Hydraulics	TAM PACKER LIST					
		Volatile Organics	3	3	1	1	8
		Extractables	3	3	1	--	7
		Metals / Inorganic ions (Field filtered)	3	3	1	--	7
		PCB's	3	3	1	--	7
		Physical chemistry	3	3	1	--	7

TABLE III-1 (Page 2 of 2)
SUMMARY OF SAMPLING AND ANALYSIS
KNOX-KERBEL MONITOR WELL

MATRIX	FIELD PARAMETERS	LAB PARAMETERS	INVESTIGATIVE SAMPLES Total No.	FIELD QUALITY CONTROL SAMPLES DUPLICATE SAMPLES	FIELD BLANKS	TRIP BLANKS	MATRIX TOTALS
Ground Water (5)	Temp, pH, Cond, DO Pump test	APP. IX INCLUSIVE PARAMETER LIST					
		Volatile Organics	2	1	1	1	5
		Extractables	2	1	1	--	4
		Metals / Inorganic ions and cyanide (filtered)	2	1	1	--	4
		PCB's / pesticides	2	1	1	--	4
		Herbicides	2	1	1	--	4
		Dioxins / furans	2	1	1	--	4
		Polar semi-volatiles	2	1	1	--	4
		Physical chemistry	2	1	1	--	4
Ground Water (6)	OVA or HNu, Temp, pH, Cond, DO	CONTINUING APP. IX PARAMETER LIST	For Ongoing quarterly sampling				
		Volatile Organics	2	1	1	1	5
		Extractables	2	1	1	--	4
		Metals / Inorganic ions and cyanide (filtered)	2	1	1	--	4
		PCB's / pesticides	2	1	1	--	4
		Herbicides	2	1	1	--	4
		Dioxins / furans	2	1	1	--	4
		Polar semi-volatiles	2	1	1	--	4
		Physical chemistry	2	1	1	--	4

1: Testing of water to be used in preparing mud prior to initiation of drilling.
2: Test aqueous phase from mud samples prior to sampling the Brassfield, Cincinnatian, Trenton and Knox with TAM Packer system; and at well TD. The Cincinnatian, Trenton and Knox Tam Packer tests are considered here as one sampling event. Since the mud in the wellbore will be homogeneous during these TAM runs, only one drilling mud sample will be collected.
3: Brassfield formation fluid sampling with TAM.
4: Formation fluids recovered via TAM from Cincinnatian, Trenton and Knox. Considered as one sampling "event", therefore only one set of blanks collected.
5: Initial sampling at well completion, from the Knox-Kerbel monitor zone. Two independent samples separated by one purged well volume. One duplicate.

TABLE III-2 (Page 1 of 1)
SUMMARY OF SAMPLING AND ANALYSIS
BRASSFIELD MONITOR WELL

MATRIX	FIELD PARAMETERS	LAB PARAMETERS	INVESTIGATIVE SAMPLES Total No.	FIELD QUALITY CONTROL SAMPLES DUPLICATE SAMPLES	FIELD BLANKS	TRIP BLANKS	MATRIX TOTALS
Drilling Mud (1)	Temp, pH, Cond, DO	BRASSFIELD LIST					
		Total organic carbon	2	2	2	--	6
		Metals / Inorganic ions (Field filtered)	2	2	2	--	6
		Physical chemistry	2	2	2	--	6
Ground Water (2)	Temp, pH, Cond, DO Pump test	BRASSFIELD LIST					
		Total organic carbon	1	1	1	--	3
		Metals / Inorganic ions (Field filtered)	1	1	1	--	3
		Physical chemistry	1	1	1	--	3
Ground Water (3)	OVA or HNu, Water level, Temp, pH, Cond, DO	BRASSFIELD LIST					
		Total organic carbon	1	1	1	--	3
		Metals / Inorganic ions (Field filtered)	1	1	1	--	3
		Physical chemistry	1	1	1	--	3

1: Test aqueous phase of mud when long string casing point is reached an after coring the Brassfield.
2: Initial sampling of the Brassfield monitor zone at well completion.
3: Brassfield monitor zone ongoing semi-annual sampling.

FIGURE III-1

KNOX-KERBEL MONITOR WELL DRILLING SCHEDULE

TASK

1) Prepare location
2) Drill conductor hole, set and cement conductor
3) MI & RU drilling rig
4) Drill and core surface casing hole to 780 ft
5) Run logs
6) Run, cement and test surface casing
7) Drill to top of Kerbel, run logs, test Cincinnatian, Trenton and Knox
8) Run and cement long string casing, test casing
9) Core Knox-Kerbel, log and test
10) Develop monitor zone
11) Complete well-(run packer and sampling tubing, piezometer tubing, pressure test annulus, install pump and motor, install transducer)
12) Run step rate pump test (SRPT)
13) Purge and produce well for sampling
14) Move out rig, clean location

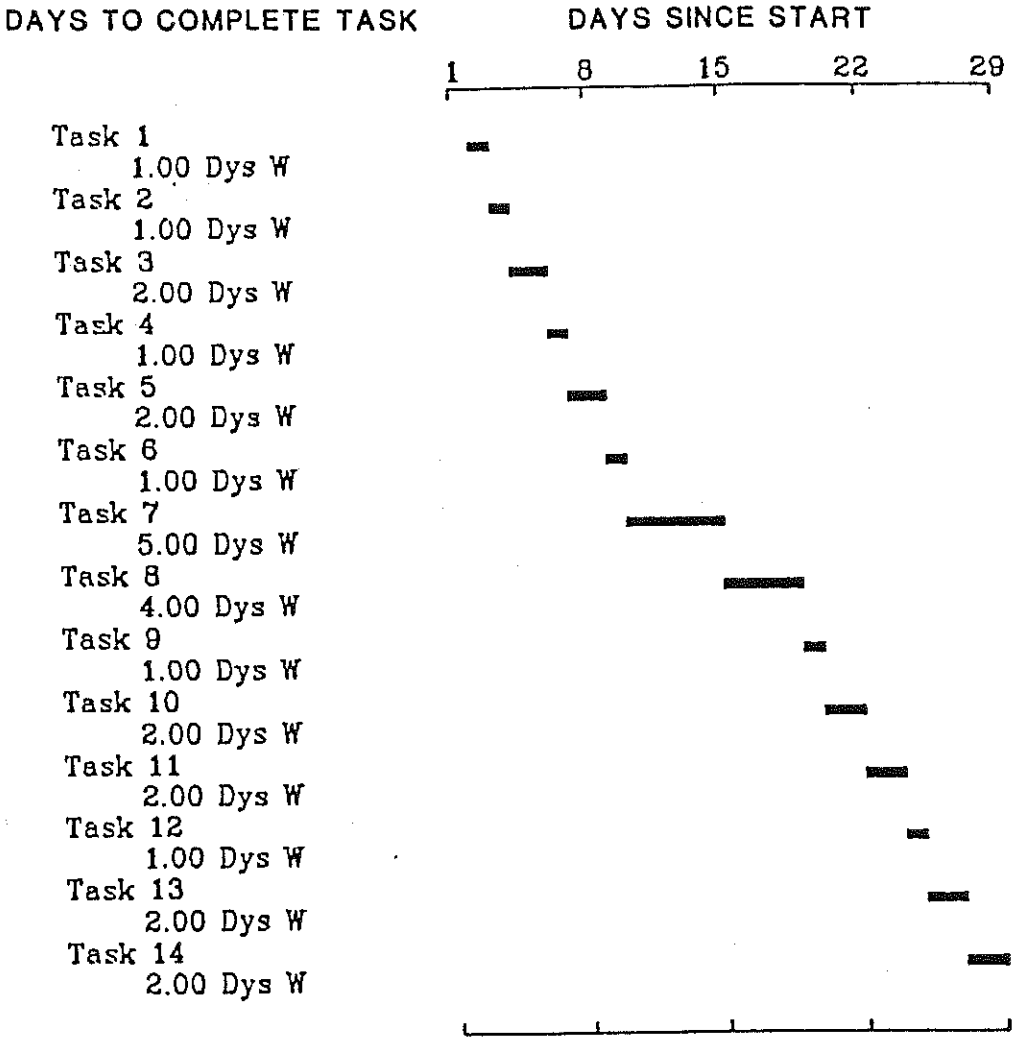
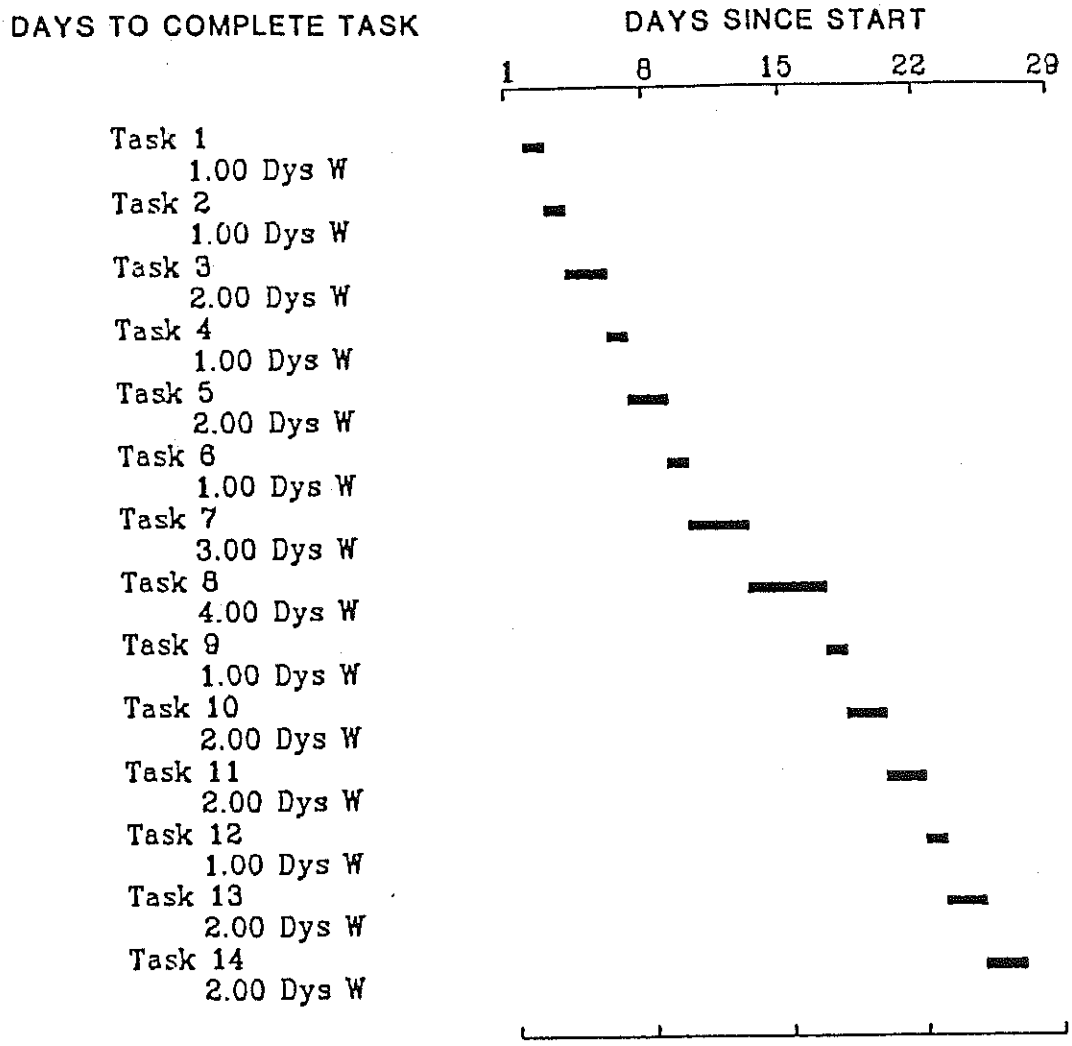


FIGURE III-2

BRASSFIELD MONITOR WELL DRILLING SCHEDULE

TASK

1) Prepare location
2) Drill conductor hole, set and cement conductor
3) MI & RU drilling rig
4) Drill surface casing hole
5) Run logs
6) Run, cement and test surface casing
7) Drill to top of Brassfield, run logs,
8) Run and cement long string casing, test casing
9) Core Brassfield, log and test
10) Develop monitor zone
11) Complete well-(run packer and sampling tubing, pressure test annulus, install pump and motor
12) Run step rate pump test (SRPT)
13) Purge and produce well for sampling
14) Move out rig, clean location



IV. PROJECT ORGANIZATION AND RESPONSIBILITY

The GWMP to be conducted for Chemical Waste Management, Inc's Vickery facility has been designed and coordinated under the overall supervision of Texas World Operations, Houston, Texas, with continuing input from USEPA Region V. Texas World Operations will maintain supervision and coordination of the GWMP during the well installation, initial testing, analysis, interpretation and reporting stages.

Sub-contractors to supply the drilling rig, drilling tools, ancillary equipment, rig operational personnel, geophysical logging services and mud logging have not yet been selected. These selections will be made following approval of a GWMP and QAPP by USEPA and OEPA. Data regarding sub-contractor selection will be provided to USEPA and OEPA prior to the start of drilling operations.

Field testing procedures for chemical parameters of mud and recovered formation fluids will be carried out by Texas World personnel. Hydrological testing of the various subsurface formations will be performed by Texas World in conjunction with sub-contractors such as TAM International, who will supply specialized downhole equipment.

All petrophysical and petrographic testing conducted on cores recovered from the monitoring wells will be will be carried out by OMNI Services, Inc., in their Houston, Texas laboratory facility. A representative of OMNI Services will be present during core recovery

operations to ensure proper handling and preservation of the core prior to shipment to the laboratory.

Drilling mud and formation fluid sampling performed during well installation and initial monitor zone testing will be conducted by Texas World's field personnel.

All laboratory chemical analyses performed in conjunction with the monitor well's installation and initial sampling will be performed by Enseco, Inc., under Enseco quality assurance/quality control procedures approved and supervised by WMI Environmental Monitoring Laboratories, Inc., a subsidiary of Waste Management, Inc., and Texas World. All chemical analytical work will be performed at Enseco-Rocky Mountain Analytical Laboratory (RMAL) in Arvada, Colorado. Enseco's address and phone numbers are provided below.

Enseco - Rocky Mountain Analytical Labortory
4955 Yarrow Street
Arvada, Colorado 80002
Phone (303) 421-6611
Facsimile (303) 431-7171

Preliminary data assessments will be performed by the testing facility performing a particular set of analyses. Final data assessment of all information gathered during the course of the comprehensive well installation and testing program will be performed by Texas World Operations. Data validation will be performed by QualtaLex, Inc., Lakewood,

Colorado.

Texas World Operations will maintain close contact with Chemical Waste Management, Enseco, WMI Environmental Monitoring Laboratories, QuantaLex, TAM International, OMNI Services, additional sub-contractors as required, USEPA Region V and OEPA personnel throughout the course of the GWMP implementation in order to effectively coordinate the overall testing program. All relevant personnel will be kept informed of the progress of drilling and testing, preliminary results, and any changes in procedures which may be mandated by initial test results.

Texas World Operations is responsible for internal Performance and System Audits of sample collection and field measurements. WMI Environmental Monitoring Laboratories is responsible for internal Performance and System Audits of laboratory analysis. USEPA Region V Central Regional Laboratory (CRL) is responsible for external performance and system audits of the Enseco laboratory, and Region V CRL and/or Central District Office (CDO) are responsible for external performance and system audits of field activities.

Table IV-2 presents an outline of project organization and responsibility. Figure IV-1 is a generalized organization chart for the project.

TABLE IV-1

PROJECT ORGANIZATION AND RESPONSIBILITY

MANAGEMENT ORGANIZATION

CWM - Project Manager
CWM - Site Manager
Texas World - Project Manager
Texas World -Site Manager
Sub-contractors - Site Representatives

USEPA - Project Coordinator
OEPA - Project Coordinator

QUALITY ASSURANCE ORGANIZATION

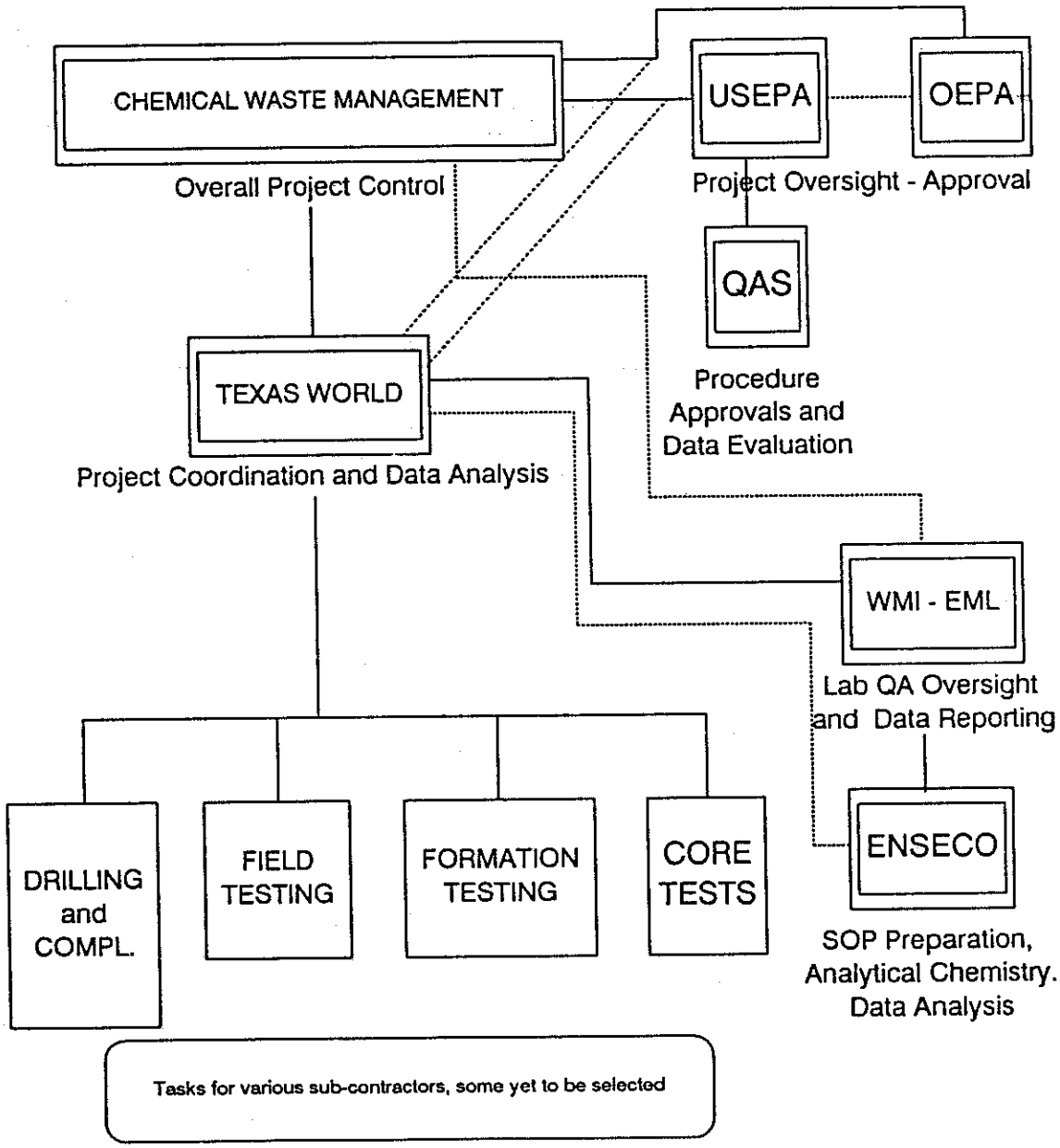
Tasks	Responsible Party
Final approval of GWMP/QAPjP	USEPA Region V UIC Project Coordinator USEPA Region V Quality Assurance Officer OEPA Quality Assurance Officer
QA review and approval of GWMP/QAPjP, reports, field and lab SOPs and procedures	CWM Project Manager
QA review of reports, SOPs, field activities and procedures	Texas World Project Manager
Internal performance and system audits of field activities and procedures	Texas World Site Manager
QA review and approval of analytical chemical laboratory reports, sops. Internal performance and system audits of laboratory procedures	Environmental Monitoring Laboratories Manager of QA Programs
External performance and system audits of field procedures	USEPA Region V Project Coordinator and Region V CRL\CDO

TABLE IV-1 continued

Tasks	Responsible Party
External performance and system audits of laboratory procedures	USEPA Region V CRL and OEPA QA Officer
Data validation	QuantaLex, Project Manager
Final evidence file maintenance	CWM Vickery, Project Manager
FIELD OPERATIONS	
Drilling and well installation, field testing and measurements	Texas World Site Manager, who supervises various sub-contractors for drilling, logging and testing operations
Sample collection	
TAM Packer tests	Texas World/TAM Site Managers
Monitor zone sampling	Texas World Site Manager
Core recovery, field preservation	OMNI field representative
LABORATORIES	
Initiation of requests	Texas World Project Manager
Analytical chemistry	Enseco, Laboratory Director of QA
Petrophysical/petrographic analysis	OMNI, QA Manager

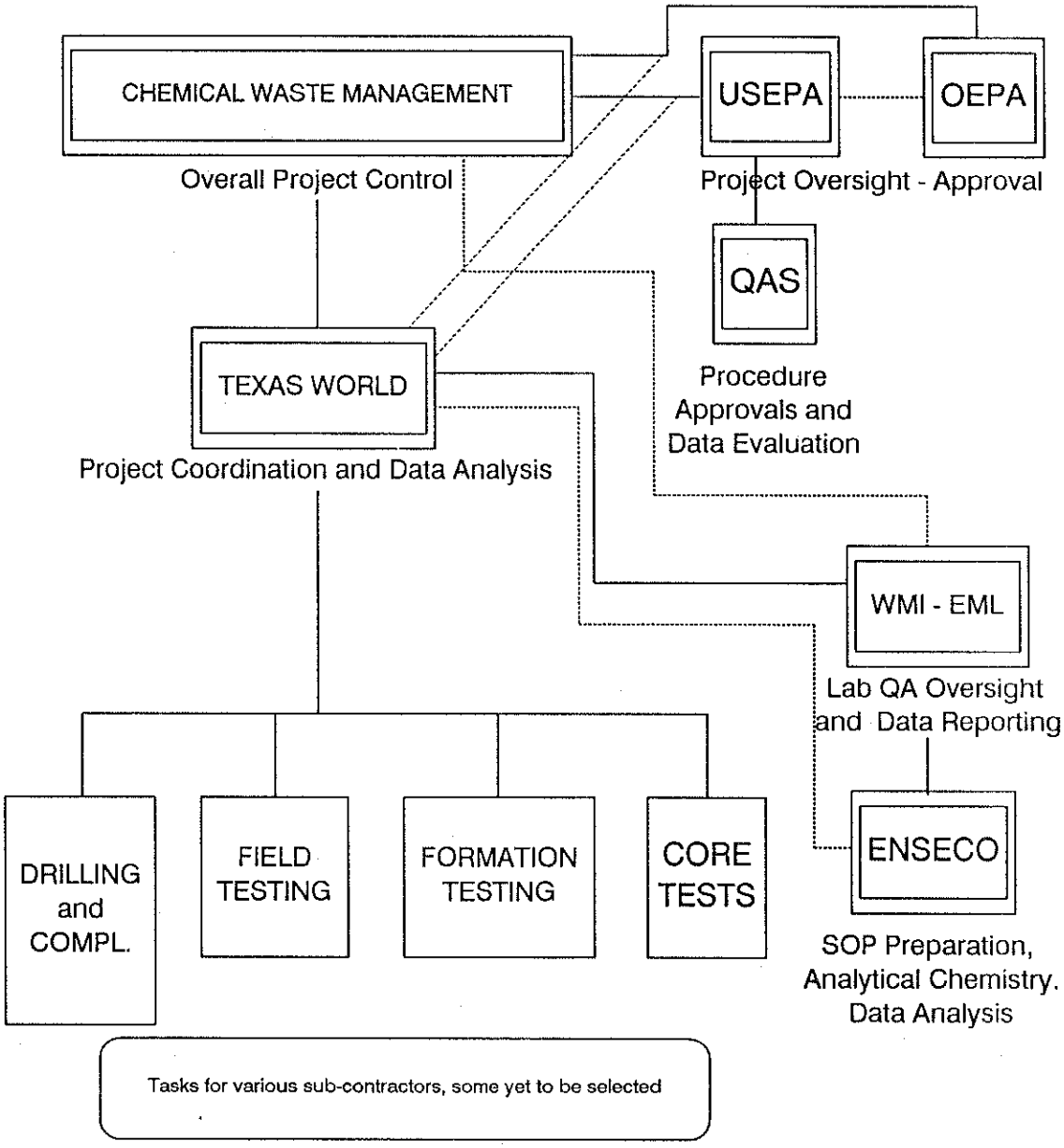
GWMP ORGANIZATION CHART

FIGURE IV-1



GWMP ORGANIZATION CHART

FIGURE IV-1



V. QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY

1. LABORATORY MEASUREMENTS

This section describes the assessments WMI Environmental Monitoring Laboratories (EML) has established to measure compliance with accuracy, precision, completeness, representativeness, and comparability goals for resultant laboratory data. Data is required to meet or exceed the criteria specified in this QAPjP for each applicable assessment parameter. Several different tools, including audits and performance evaluation programs, assist the EML in determining the level of compliance with these objectives. The QA objectives in specific terms are outlined below.

1.1 ACCURACY

"Accuracy" defines the relationship between the laboratory's measurement of a sample's concentration and the "true", but unknown concentration of the sample. However, because the "true" concentration in a sample is unknown, accuracy of sample results must be indirectly measured. This assessment is accomplished using percent recovery of matrix spike determinations to assess matrix accuracy, and percent recovery of Quality Control Standards to assess laboratory accuracy.

Percent recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{SS - US}{S} \times 100$$

where: SS = Concentration of the analyte in the spiked sample;
US = Concentration of the analyte in the unspiked sample;
S = Concentration of the analyte added to the sample.

Control criteria for percent recovery are taken from regulatory method requirements and/or historic laboratory performance, as established by the applicable regulatory method(s). The laboratory's goal is to comply with all applicable criteria. Project acceptance control limits for accuracy are specified in QAPjP Appendix A-11.

1.2 PRECISION

"Precision" is the assessment of the variability that can be expected in data that result from the procedures employed. It provides a measure of consistency or reproducibility. Typically it is estimated through duplicate measurements of a single sample or standard. The procedures that are assessed begin at the point the duplicate aliquot is separated. The various terms defined for duplicates delineate which conditions and procedures are included in the precision assessment. Precision is measured by using "relative percent difference" (RPD). RPD is determined as follows:

for organic analyses,

$$\text{Relative Percent Difference} = \frac{(MS - MSD)}{(MS + MSD)\,(0.5)} \times 100$$

where MS = Concentration of the analyte in the spike recovery from the matrix spike

MSD = Concentration of the analyte in the spike recovery from the matrix spike duplicate

and for inorganic analyses,

$$\text{Relative Percent Difference} = \frac{(S - SD)}{(S + SD)\,(0.5)} \times 100$$

where S = Concentration of the analyte in the standard or sample

SD = Concentration of the analyte in the standard or sample

Control criteria for RPD are taken from regulatory method requirements and/or historic laboratory performance, as established by the applicable regulatory method(s). The laboratory's goal is to comply with all applicable criteria. Project acceptance control limits for precision are specified in QAPjP Appendix A-11.

1.3 COMPLETENESS

"Completeness" defines the percentage of valid data obtained from a procedure compared to the amount that was expected to be obtained under

correct normal conditions. It is the amount of data reported to the data user without accompanying "qualification" of the data's validity. The goal is to produce 100% usable data; however, a number of factors beyond project control (for example, the effect of sample matrix) may adversely effect this objective.

The percent completeness of analysis results may be estimated through review of result data and associated data qualifications. Data qualifiers are explanatory information associated with particular results that informs the data user of any unusual circumstances of analysis or limitations of the result data. Based on the data quality objectives of the project, a data qualifier may or may not invalidate a result. The affect a data qualifier has on the validity of a result is tabulated, as is the total number of reported results. The percent of invalid result data is the measure used for estimation of the completeness. Project acceptance control limit for completeness is 95 percent.

1.4 REPRESENTATIVENESS

"Representativeness" defines the extent to which the results determined for a particular sample point accurately reflect the sample point's composition.

The EML has instituted a number of steps to ensure representative results.

These steps include:

- Development and implementation of the WMI Sampling Manual, which defines the steps required during sample collection.
- Sample team training, which demonstrates proper technique with collecting field samples.
- Sample team audits to assess the compliance of the sampling teams with established policy.
- The Aquapak System and Bottle Evaluation Program that eliminate sources of sample contamination.
- Use (where feasible) of dedicated sampling equipment at each WMI/CWM monitoring well.
- The field duplicate program, in which two samples taken from the same location at the same time are submitted and analysts are unaware which samples are duplicates.
- Requirements that analysts thoroughly mix samples before aliquoting them.
- Historical review of data from each ground water well.
- Requirements that the time from sample collection to either the initiation of analysis or sample extraction not exceed the allowed holding time specified for the project.

Where replicate samples are taken at a particular location, the extent to which the results correlate can be used to estimate the representativeness of the overall sampling and analysis process in so far as precision can estimate representativeness. Also, laboratory matrix precision can be compared to this overall precision to evaluate the contribution of the sampling component to the overall imprecision, and representativeness.

1.5 COMPARABILITY

"Comparability" defines the extent to which results are consistent, both from batch to batch, and with results the rest of the laboratory community produces. For example, if the EML were to analyze the same sample, using the same procedure, on two different days, the results should be comparable. Likewise, two laboratories, adhering to similar methods and quality control procedures, should produce comparable results for identical samples. EML demonstrates comparability through the following steps:

- Requiring the use of calibration standards traceable to NIST. Documentation verifying this must be maintained with the standard preparation information. Only where NIST-traceable standards can not be obtained will the laboratory use other certified sources.
- Requiring that all calibrations be verified with a standard from a certified source different than the calibration standard source.
- Requiring the use of regulatory agency-approved methods for all analyses.
- Requiring participation in inter-laboratory performance evaluation programs, including the EPA Water Pollution and Water Supply programs, and other state sponsored inter-laboratory programs.
- The field duplicate program and the historical review of current ground water well data with prior data from the same well.

As an estimate of comparability, tabulation of the acceptable inter-laboratory comparison results reported relative to the total number reported may be used. A goal of 95% acceptable results on inter-laboratory comparison analyses for comparability is routinely used.

1.6 QUALITY CONTROL MEASUREMENT REQUIREMENTS

The quality control measurements routinely conducted for each method of analysis are specified in laboratory SOPs and Quality Program documents, and are measures that meet or exceed the requirements of the applicable regulatory methods. The control criteria (limits developed from historical data or those specified in regulatory methods) must be at least as stringent as those required by regulatory agencies and methods. Further, corrective actions implemented when an analysis does not meet these criteria must be documented. Any additional quality control measures, greater frequency of measures, or different control criteria beyond those specified in laboratory policy, Methods and SOPs are delineated in this QAPjP, and must be adhered to in analysis of project samples.

2. FIELD MEASUREMENTS

2.1 pH, CONDUCTIVITY, TEMPERATURE AND DISSOLVED OXYGEN

Chemical Waste Management will insure the accuracy of field measurements for conductivity, pH, temperature and dissolved oxygen through thorough pre-measurement calibration of the field instrumentation, and post-measurement verification. Manufacturer's recommendations on instrument calibration will be followed.

Field measurements of conductivity, pH, temperature and dissolved oxygen

during well purging and sampling will be performed through the usage of a flow cell. A representative split of the fluids pumped from the well will be directed through the flow cell by means of a diverter valve to reduce instrument degradation due to excessive volumes of possibly abrasive fluid passing the sensors.

The pH measurement instrument will be checked for accuracy by performing two measurements on three standard buffer solutions, following calibration. Each measurement will be within ±0.1 pH units of the standard. Precision will be assessed through replicate measurements. The standard deviation of four replicate measurements must be less than or equal to 0.2 pH units. The instrument will be withdrawn, rinsed with deionized water and re-immersed between each replicate measurement. The calibration and verification will be done in the field before well purging begins and after the last environmental sample has been collected. Resolution of the instrument used will be less than or equal to 0.02 pH units as per manufacturer's specifications, and range will be at least from 0 to 14 pH units.

Conductivity measurements will be converted to an equivalent at 25 °C (specific conductance) with a conductivity meter with a built in temperature correction factor. Conductivities considerably higher than that commonly found in shallow ground water monitoring wells are expected to be encountered in the fluids

from the tested formations. A conductivity meter with a range of 0 to 199,990 umhos/cm will be used. Accuracy shall be within 5% of a known standard. Precision shall be within a deviation of ±20% Relative Percent Difference as determined from four measurements of a known standard.

Temperature will be measured using a thermometer on the conductivity meter with a range of at least -2 to 50 °C. Accuracy will be to within ±0.5 °C and will have been calibrated against a known standard.

The dissolved oxygen meter will function through a range of at least 0 to 50 mg/l of oxygen. Accuracy shall be within ±1.0% of full scale. The instrument will automatically correct for temperature, salinity and barometric pressure factors.

2.2 ORGANIC VAPOR MONITORING

Upon opening the well caps prior to any periodic purging and sampling event, the air in the wellheads will be tested for organic vapors to guard against possible fire, explosion or toxicity hazards. Either an organic vapor analyzer or a photoionization chamber will be used. Any concentration of organic vapors present will be measured with an accuracy of 1 % of full meter scale and resolution of less than 1 ppm.

2.3 PRESSURE MEASUREMENTS

A 50 psig range pressure transducer has been selected for installation in the Knox-Kerbel well at a depth approximately 50 feet below the static water level in order to continuously monitor the formation pressure. Hanging the transducer at approximately 50 feet below the water level allows the instrument to function near the midpoint of its range for increased accuracy. The selected instrument will operate with a combined repeatability and hysteresis error of less than 0.25% of full scale. The pressure transducer is vented to the atmosphere via nylon tubing in the cable to allow automatic compensation for barometric pressure changes. The electrical output from the transducer is compensated for temperature in the range of 0 to 40 °C. The signal from the transducer will be recorded in the CWM control room for monitoring and incorporation into periodic reports as specified in Section 10 of the GWMP.

For at least one month after well installation, the barometric pressure at the site will be recorded independently with a barometer. The barometer will have an accuracy of ±1.0 mb. If calculations indicate the vented transducer in the well is adequately compensating for barometric variations, the independent barometric pressure monitoring may be discontinued.

Once a month following installation, the depth to water in the Knox-Kerbel

well will be established using an electric tape (measured to ±0.1 ft). The static water level will be referenced to msl utilizing a control point on the wellhead established by licensed surveyor to within 0.01 feet. The pressure transducer readout will be converted to head and msl using the specific gravity of the fluid last sampled from the well for the conversion. A comparison of the two static water levels will be made. The transducer will then be raised a measured distance in the well (approximately 10 feet) and its signal allowed to stabilize. The change in measured pressure will be calculated. When compared to the known distance the instrument was raised, this will enable a calculation of the specific gravity of fluid in the hole to be made and compared to that measured during the last sampling event.

Should the static water level calculated from the pressure transducer readout vary from that measured with the electric tape by more than 5 feet, the electric tape measurement and transducer calibration will be repeated. Should the repeat measurements still vary by more than 5 feet, the pressure transducer will be removed from the well for recalibration or replacement.

VI. SAMPLING PROCEDURES

1. SAMPLE POINT SELECTION

CWM has proposed, and the USEPA Region V and OEPA UIC Sections have agreed to, the installation of two monitor wells at the Vickery, Ohio facility to monitor for no-migration out of the injection zone and to monitor ground water quality in the lowermost USDW. The deeper monitor well will be drilled and completed in the Knox-Kerbel Formations at ±2410-2440 ft prior to the drilling and completion of the shallower monitor well, proposed to be completed in the Brassfield Formation at ±700-720 ft. See Figures 2-1 and 2-6 in the GWMP for a graphical presentation of the stratigraphic intervals to be completed, and their relationship to the injection interval and USDW. Both of the intervals to be monitored must meet the requirements of the USEPA Region V monitoring plan guidelines (GWMP Appendix D), recommendations (GWMP Appendix C) and OEPA permit requirements (GWMP Appendix B) for stratigraphic location, aerial extent, zone thickness and permeability.

During the course of drilling the monitor wells, openhole tests of reservoir pressure will be conducted in the Brassfield, Cincinnatian, Trenton and Knox Formations using the TAM packer system. Formation fluid recovered from the Brassfield will be tested for TDS only. Fluid recovered from the Cincinnatian, Trenton and Knox will be tested for the TAM packer list of chemical and physical parameters. These particular zones have been selected for testing in order to provide data on the site's vertical hydraulic gradient,

petrophysical parameters to verify modeling input, and the evaluate the possibility of the existence of contamination from prior releases of injectate.

The Knox-Kerbel Formation monitor well will be located to the north-northeast, and within 100 ft of CWM's existing Disposal Well No. 2. The Brassfield Formation monitor well will be located to the east, and within 100 ft of CWM's existing Disposal Well No. 2. These locations are near the current and historical center of injection at the CWM site and near a presently active injection well. These proposed locations offer sufficient operational space for the safe installation and operation of the wells, avoiding as much as possible the existing infrastructure of the CWM facility, including a high pressure pipeline to the northwest of the No. 2 well. The exact location will be submitted with the final completion report. See Figures 2-1 and 2-4 of the GWMP for a plan view of the approximate location of the proposed monitor wells in relation to the existing injection wells at the facility. Additional information on siting was presented in Section 2 of the GWMP.

2. SAMPLE MATRICES AND SAMPLING METHODS

Three matrices will be sampled during the course of drilling and completing the monitor wells, initial monitor zone sampling and ongoing quarterly or semi-annual sampling. These are: 1) make up water for drilling mud, 2) drilling mud, 3) groundwater. A summary of sampling and analysis requirements was previously presented in Tables III-1 and III-2.

2.1 MAKE UP WATER

Native drilling mud will be used during the installation of both monitor wells. This mud is formed by the reaction of the drilled formation material with additional water. The water added to the hole during drilling will be obtained from the truck wash well at the Vickery site, and transported to the drilling location by tank truck. This make up water will be sampled from the tank truck at the drill site prior to spudding the first well. Water will be drawn directly through the truck's outlet valve into appropriate sample bottles for parameters on the TAM Packer list not requiring field filtering. Samples that require field filtering will be passed through an in-line .45 micron filter prior to emplacement in appropriate sample containers. Appendix A-1 (an excerpt from the Waste Management Inc, Manual for Groundwater Sampling, 1991, which will be referenced as WMIMGS later) details the parameters which do and do not require field filtering. Temperature, specific conductance, pH and dissolved oxygen will be measured in the field. Sample containers, preservation, and technical holding time requirements are addressed in QAPjP Appendix A-4. Field duplicate samples will be collected as described in QAPjP Tables VI-1 and VI-2. Sample preservation, handling and chain of custody methodology are further detailed or referenced later in this section.

2.2 DRILLING MUD

The drilling mud, composed of drilled formation materials and make up

water, will be tested periodically as indicated in Tables III-1 and III-2 for the TAM Packer parameter list. Grab samples will be taken directly from the mud circulation system at the point of discharge from the shale shaker. Field measurements of temperature, specific conductivity, pH and dissolved oxygen will be taken. Samples for volatile and semi-volatile analysis will be emplaced directly into appropriate sample containers. The total mud sample (liquid plus solids portions) will be subjected to analysis for volatiles and semi-volatiles. Mud for other parameter analysis will be collected in a plastic lined container, sealed, and allowed to settle for 12 hours. The aqueous portion of the mud will be decanted, dipped or siphoned as appropriate into a .45 micron pressure filter prior to emplacement in sample containers. Sample containers, preservation, and technical holding time requirements are addressed in QAPjP Appendix A-4. Field duplicate samples will be collected as described in QAPjP Tables VI-1 and VI-2. Sample preservation, handling and chain of custody methodology are further detailed or referenced later in this section.

While the wells are being drilled, and footage is actually being made, mud samples will be collected at the shale shaker every four hours to check organic vapor concentration. Approximately one-half quart of mud will be collected in a one quart glass container, sealed and allowed to set for 15 minutes. The headspace in the container will then be checked for organic vapor concentration with a

photoionization meter or organic vapor analyzer. Measurements will be reported to and recorded by the Texas World Site Supervisor.

2.3 GROUNDWATER

2.3.1 TAM Packer Recovered Samples

The TAM packer tool is designed to isolate a subsurface zone with inflatable packers, and using a down hole pump powered from the surface the tool is able to produce the zone into an isolated sample collection section. Four one liter sample bottles are available for filling with each wireline run to obtain formation fluid samples. For the Brassfield Formation test, sufficient fluid will be recovered for TDS determination only. Once the formation fluid samples have been recovered at the surface, the recovered fluid will be slowly emptied directly into appropriate sample bottles for parameters on the TAM Packer list not requiring field filtering. Samples that require field filtering will be passed through a .45 micron pressure filter prior to emplacement in appropriate sample containers. Appendix A-1 indicated the parameters which do and do not require field filtering. Temperature, specific conductance, pH and dissolved oxygen will be measured in the field. Sample containers, preservation, and technical holding time requirements are addressed in QAPjP Appendix A-4. Field duplicate samples will be collected as described in QAPjP Tables VI-1 and VI-2, and in 8.3 of this section. Sample preservation, handling and chain of custody methodology are further detailed or

referenced later in this section.

2.3.2 Samples Recovered Using Dedicated Sampling Equipment

Fluid samples from the permanent monitoring zones (Knox-Kerbel and Brassfield) will be delivered to the surface by the rod operated pumps to be installed in the wells. Water will be directly emplaced into appropriate sample bottles for parameters on the Appendix IX parameter list not requiring field filtering. Samples that require field filtering will be passed through an in-line .45 micron filter prior to emplacement in appropriate sample containers. Appendix A-1 indicated the parameters which do and do not require field filtering. Temperature, specific conductance, pH and dissolved oxygen will be measured in the field. Sample containers, preservation, and technical holding time requirements are addressed in QAPjP Appendix A-4. Field duplicate samples will be collected as described in QAPjP Tables VI-1 and VI-2, and in 8.3 of this section. Sample preservation, handling and chain of custody methodology are further detailed or referenced later in this section.

3. SAMPLING PROTOCOL

3.1 TAM PACKER TESTS

During the course of drilling the Knox-Kerbel monitor well, fluid samples will be recovered from the Brassfield Formation, Cincinnatian Series, Trenton and

upper Knox Formations utilizing the TAM packer system. The purpose of the tests conducted with the TAM wireline tester is to obtain accurate initial reservoir pressures, representative samples of native formation fluids, and formation permeability and skin factor. The test design, equipment and procedures outlined in the GWMP have been specifically designed to achieve these goals. An engineer familiar with test interpretation will be on site to ensure that the required samples and pressures have been collected and that the test goals have been achieved.

The TAM packer tool is designed to isolate a zone with inflatable packers using a down hole pump powered from the surface. The tool is able to produce the zone, measure pressure, temperature, resistivity, and pH downhole to ensure that parameter stabilization has occurred prior to capturing a representative formation fluid sample. A fluid volume of at least three times the volume contained within the packed off interval of the wellbore will be purged prior to capturing any environmental samples. The samples of the fluid are stored in the tool's sample containers for later chemical analysis at the surface. Since fluid properties are measured downhole and transmitted to the surface in real time, a representative sample can be reliably obtained. An advantage as compared to the conventional DST is that the sample fluid is not exposed to possible contamination from the drill pipe as it is swabbed or bailed to surface. A description of the TAM packer apparatus, tool specifications and detailed test procedures were previously

supplied in Appendix G of the GWMP.

3.2 KNOX-KERBEL AND BRASSFIELD MONITOR ZONES

3.2.1 Well Purging

Prior to purging the well or collecting any samples the air in the wellhead will be tested for organic vapors using either a organic vapor analyzer or a photoionization analyzer to guard against possible fire, explosion or toxicity hazard. Measured values will be recorded.

CWM plans to purge at least three well volumes of fluid prior to initiating collection of samples for analysis. CWM will monitor pH, conductivity, temperature and dissolved oxygen content via an in-line flow cell during purging until those parameters have stabilized to the extent possible based on the site specific hydrogeologic characteristics. "Stabilization" is defined here as the point at which the slope of the plots of three monitored parameters (y axis) versus purged fluid volume (x axis) approaches zero, and at least three well volumes of fluid have been produced. The volume of fluid purged will be measured with an in-line flow meter located immediately downstream of the well's fluid outlet, and upstream of any diverter valves, flow cells or filters which may be in place. This will insure that all produced fluid for each purging and sampling event is properly measured and recorded

Equipment used in monitoring the field parameters will be calibrated to manufacture's specifications prior to each purging and sampling event. Parameter values will be plotted versus produced fluid volume during purging. Suitable rates for purging the well will have previously been established through the performance of the SRPT performed following well development.

The wells will be purged using a rod operated positive displacement piston pump run in the hole on 1 1/2 inch diameter stainless steel tubing inside the 2 7/8 inch sampling tubing. This type of pump enables purging to proceed at a rate of up to 4 gpm during the initial stages, then slow down the fluid production rate while the last well volume is being removed. This will minimize turbulence, degassing or aeration of produced liquids, delivering representative fluid samples to the surface. The 1 1/2 inch stainless steel tubing leading from the pump to the surface will actually be completely full of fluid during purging and sampling, and there will be no opportunity for the produced fluid to mix with air in the well above the pump. Actual fluid production rates during sampling will be approximately 100 ml/min, supplied with the same pump used for purging.

The excess fluid produced by pumping the monitoring well during purging and sampling procedures will be temporarily stored in appropriate tankage, and ultimately be disposed of into one of the active injection wells at the site or by

alternative disposal methods.

3.2.2 Measurements During Well Purging

During well purging, pH, conductivity, temperature and dissolved oxygen content will be continuously monitored utilizing an in-line flow cell. Measured values will be plotted versus produced fluid volume. Excessive wear and abrasion on the measuring probes and the flow cell (and possible measurement errors) can be caused by the throughput of large volumes of produced fluids during purging and sampling. To prevent this, the flow cell will be installed downstream of a diverter valve which can direct a reduced volume of produced fluid to the cell. The flow cell will monitor a representative split of the produced fluid with reduced danger of instrument degradation from wear. All produced fluids will be retained in appropriate tankage for later disposal.

3.2.3 Determination of Well Volume

The Knox-Kerbel and the Brassfield monitor wells will be tubing and packer assembly type completions. Well volume will be calculated using the following formula:

Well Volume = [(PD-SWLD) x CSAT] + [(TWD - PD) x CSAO]

where

SWLD = static water level depth
TWD = total well depth
PD = packer depth
CSAT = cross sectional area of tubing
CSAO = cross sectional area of open hole

3.2.4 Sampling Rates

The rod operated pumping mechanism to be installed in the monitor wells is capable of delivering fluid to the surface at a variable rate. When actually sampling the wells for analytical chemistry purposes, the fluid production rate will be slowed down to approximately 100 ml/min. This rate will facilitate the filling of appropriately sized sample containers directly from the sampling tubing outfall while minimizing turbulence, degassing or aeration of the samples which could adversely affect laboratory results. The rod operated positive displacement pump to be used in each well is uniquely suited for both sampling and purging requirements with a minimum of sample disturbance.

3.2.5 Field Measurements (pH, Conductivity, Temperature and Dissolved Oxygen)

Fluid conductivity, pH, temperature and dissolved oxygen content measurements will be monitored continuously in the field during both purging and sampling by means of an in-line flow cell at the outfall from the stainless steel

sampling tubing. These parameters are extremely vulnerable to alteration after production from the well, and are best measured in situ. The use of an in-line flow cell and associated probes will insure that the fluid samples do not contact the atmosphere prior to field parameter measurement.

Conductivity at field measured temperature will be converted to an equivalent at 25 °C. Conductivity, temperature, pH and dissolved oxygen measurements will be recorded at the beginning and end of the sampling period. These parameters will have been previously measured during purging operations and plotted versus cumulative fluid production volume.

3.2.6 Sample Emplacement in Containers

The exit end of the in line flow cell for measuring pH, temperature, conductivity and dissolved oxygen will be fitted with a short length of flexible Teflon tubing to facilitate filling sample containers with fluid. The containers will be filled slowly to avoid agitation, degassing or aeration of the fluid during fill up. This length of tubing will be replaced or cleaned between container fillings where appropriate to prevent cross contamination of samples. Samples requiring filtration to .45μ will be filtered by use of a disposable in-line filter cartridge downstream of the flow cell. Additional detail on filling sample bottles is found in Appendix A-2

(from WMIMGS). Filled sample containers will be placed in an AquaPak for cooling to less than 4 °C.

4. SAMPLE PACKAGING, HANDLING, SHIPMENT, FILTERING

CWM will follow the procedures and methods described in the Waste Management Inc., Manual for Groundwater Sampling (1991), several excerpts from which are included in Appendix A of this QAPjP.

4.1 SAMPLE PACKAGING

Please refer to items 4.2.1.3 and 4.4.2 in Appendix A-3 discussing usage of the AquaPak sample container, and sample bottles.

4.2 HANDLING AND SHIPMENT

Please refer to items 4.4.3.1 in Appendix A-3.

4.3 TIME CONSIDERATION

Please refer to item No.9 in 4.3.4 of Appendix A-2, and item 4.4.3.1 in Appendix A-3.

4.4 FIELD FILTRATION REQUIREMENTS AND PROCEDURE

Please refer to items 4.3.7, 4.3.7.1 and 4.3.7.2 in Appendix A-1.

5. CONTAINER TYPES, SIZE, PRESERVATION METHODS, HOLDING TIMES

For the various parameters proposed for analysis (see Section 7 of the GWMP or Section III of this QAPjP), Appendix A-4 lists the container type, volume required, preservative method and holding times to be utilized in assuring a representative sample of the formation fluid is obtained for laboratory analysis. Sampling containers will be new, or cleaned, and labeled by the WMI EML prior to being taken afield. Each sample container will be labeled as to the parameter to be analyzed for, sample size and preservative (if any) to be used. All sample bottles will be placed in AquaPaks and sealed prior to transport via overnight delivery service to the analytical laboratory.

6. PRESAMPLING PROCEDURES

Once a sampling event has been scheduled, a considerable amount of preplanning must be done to ensure that sampling will go smoothly. Presampling procedures include procurement and calibration of field meters and equipment, inspecting AquaPaks and sample bottles, coordination of shipping schedules with the analytical laboratory and reviewing the planned sampling procedures. All equipment necessary for the planned sampling event will be cleaned and/or decontaminated, operationally checked out and calibrated before being taken into the field. Instruments will be rechecked prior to actual field usage. Please refer to item 4.4.3.1 in Appendix A-3, and the sample container and preservation tables in Appendix A-4.

7. CHAIN OF CUSTODY PROCEDURES

Chain of custody procedures and a copy of the Field Chain of Custody Form to be used are documented in Appendix A-5, Item 4.2.2.1, which is an excerpt from the WMIMGS.

8. PREPARATION OF TRIP AND FIELD BLANKS, FIELD DUPLICATES

One of the fundamental responsibilities of the owner/operator is the establishment of continuing programs to ensure the reliability and validity of field and analytical laboratory data gathered as part of the overall GWMP. To this end, Chemical Waste Management plans to utilize trip blanks, field blanks and duplicate samples to aid in assuring and evaluating the quality of data obtained through the field sampling program and laboratory analytical procedures. The number of trip blanks, field blanks and duplicate samples was previously presented in Tables III-1 and III-2, but these same tables are given again here as Tables VI-1 and VI-2 for easier reference.

Trip blanks, field blanks and duplicate control samples provide independent checks on field sampling procedures, such as sample handling and storage. It should be noted that ground water analytical data is incomplete unless the analytical performance data (e.g., accuracy, precision, detection, and quantitation limits) are reported along with each set of results.

TABLE VI-1 (Page 1 of 2)

SUMMARY OF SAMPLING AND ANALYSIS

KNOX-KERBEL MONITOR WELL

MATRIX	FIELD PARAMETERS	LAB PARAMETERS	INVESTIGATIVE SAMPLES Total No.	FIELD QUALITY CONTROL SAMPLES DUPLICATE SAMPLES	FIELD BLANKS	TRIP BLANKS	MATRIX TOTALS
Make-up Water (1)	Temp, pH, Cond, DO	TAM PACKER LIST					
		Volatile Organics	1	1	1	1	4
		Extractables	1	1	1	--	3
		Metals / Inorganic ions (Field filtered)	1	1	1	--	3
		PCB's	1	1	1	--	3
		Physical chemistry	1	1	1	--	3
Drilling Mud (2)	Temp, pH, Cond, DO	TAM PACKER LIST					
		Volatile Organics	3	3	3	3	12
		Extractables	3	3	3	--	9
		Metals / Inorganic ions (Field filtered)	3	3	3	--	9
		PCB's	3	3	3	--	9
		Physical chemistry	3	3	3	--	9
Ground Water (3)	Temp, pH, Cond, DO Pressure, Hydraulics	TDS ONLY					
		Total dissolved solids	1	1	1	--	3
Ground Water (4)	Temp, pH, Cond, DO Pressure, Hydraulics	TAM PACKER LIST					
		Volatile Organics	3	3	1	1	8
		Extractables	3	3	1	--	7
		Metals / Inorganic ions (Field filtered)	3	3	1	--	7
		PCB's	3	3	1	--	7
		Physical chemistry	3	3	1	--	7

TABLE VI-1 (Page 2 of 2)

SUMMARY OF SAMPLING AND ANALYSIS

KNOX-KERBEL MONITOR WELL

MATRIX	FIELD PARAMETERS	LAB PARAMETERS	INVESTIGATIVE SAMPLES Total No.	FIELD QUALITY CONTROL SAMPLES DUPLICATE SAMPLES	FIELD BLANKS	TRIP BLANKS	MATRIX TOTALS
Ground Water (5)	Temp, pH, Cond, DO Pump test	APP. IX INCLUSIVE PARAMETER LIST					
		Volatile Organics	2	1	1	1	5
		Extractables	2	1	1	--	4
		Metals / Inorganic ions and cyanide (filtered)	2	1	1	--	4
		PCB's / pesticides	2	1	1	--	4
		Herbicides	2	1	1	--	4
		Dioxins / furans	2	1	1	--	4
		Polar semi-volatiles	2	1	1	--	4
		Physical chemistry	2	1	1	--	4
Ground Water (6)	OVA or HNu, Temp, pH, Cond, DO	CONTINUING APP. IX PARAMETER LIST	For Ongoing quarterly sampling				
		Volatile Organics	2	1	1	1	5
		Extractables	2	1	1	--	4
		Metals / Inorganic ions and cyanide (filtered)	2	1	1	--	4
		PCB's / pesticides	2	1	1	--	4
		Herbicides	2	1	1	--	4
		Dioxins / furans	2	1	1	--	4
		Polar semi-volatiles	2	1	1	--	4
		Physical chemistry	2	1	1	--	4

1: Testing of water to be used in preparing mud prior to initiation of drilling.
2: Test aqueous phase from mud samples prior to sampling the Brassfield, Cincinnatian, Trenton and Knox with TAM Packer system; and at well TD. The Cincinnatian, Trenton and Knox Tam Packer tests are considered here as one sampling event. Since the mud in the wellbore will be homogeneous during these TAM runs, only one drilling mud sample will be collected.
3: Brassfield formation fluid sampling with TAM.
4: Formation fluids recovered via TAM from Cincinnatian, Trenton and Knox. Considered as one sampling "event", therefore only one set of blanks collected.
5: Initial sampling at well completion, from the Knox-Kerbel monitor zone. Two independent samples separated by one purged well volume. One duplicate.

TABLE VI-2 (Page 1 of 1)
SUMMARY OF SAMPLING AND ANALYSIS
BRASSFIELD MONITOR WELL

MATRIX	FIELD PARAMETERS	LAB PARAMETERS	INVESTIGATIVE SAMPLES Total No.	FIELD QUALITY CONTROL SAMPLES DUPLICATE SAMPLES	FIELD BLANKS	TRIP BLANKS	MATRIX TOTALS
Drilling Mud (1)	Temp, pH, Cond, DO	BRASSFIELD LIST					
		Total organic carbon	2	2	2	--	6
		Metals / Inorganic ions (Field filtered)	2	2	2	--	6
		Physical chemistry	2	2	2	--	6
Ground Water (2)	Temp, pH, Cond, DO Pump test	BRASSFIELD LIST					
		Total organic carbon	1	1	1	--	3
		Metals / Inorganic ions (Field filtered)	1	1	1	--	3
		Physical chemistry	1	1	1	--	3
Ground Water (3)	OVA or HNu, Water level, Temp, pH, Cond, DO	BRASSFIELD LIST					
		Total organic carbon	1	1	1	--	3
		Metals / Inorganic ions (Field filtered)	1	1	1	--	3
		Physical chemistry	1	1	1	--	3

1: Test aqueous phase of mud when long string casing point is reached an after coring the Brassfield.
2: Initial sampling of the Brassfield monitor zone at well completion.
3: Brassfield monitor zone ongoing semi-annual sampling.

8.1 TRIP BLANKS

The Chemical Waste Management GWMP provides for the routine collection and analysis of quality control trip blanks. Each time a group of bottles is prepared for use in the field (a sampling event), one 40 ml trip blank vial will be included in each shipping container used for volatile organics samples. These vials are filled with deionized water by the laboratory suppling the sample bottles. (WMI-EML will supply the sample bottles and Aqua Paks, and therefore, the VOA trip blanks.) The vials filled with the blank will be transported to the sampling location and returned to the laboratory in a manner identical to the handling procedure used for the samples. These trip blanks will be subjected to the same volatiles analysis as the ground water. The trip blank samples are used to check for any cross-contamination through the vial's septa during sample shipment and storage. The concentration levels of any contaminants found in the trip blank should not be used to correct the ground-water data. The contaminant levels should be noted, and if the levels are within an order of magnitude when compared to the field sample results, re-sampling of the ground-water will be performed.

The results of the analysis of the blanks should not be used to correct the ground-water data. If contaminants are found in the blanks, the source of the contamination will be identified and corrective action, including re-sampling, will be initiated.

8.2 FIELD BLANKS

Field blanks will be prepared in the field during the sampling event, and consist of filling one sample container with the deionized water being used in the field which has been routed through the sampling device, including the field filtering apparatus. Field blanks will be collected at the frequency of one for each ten or fewer investigative samples. (See QAPjP Tables VI-1 and VI-2.)

8.3 INDEPENDENT SAMPLES, DUPLICATE SAMPLES AND BASELINE CHEMISTRY

To establish baseline chemistry, sixteen (16) independent samples collected from the Knox-Kerbel zone during the first eight (8) quarterly sampling events will be utilized. The baseline establishment period will therefore be an twenty-four (24) month time frame. These sixteen samples will be obtained during the scheduled quarterly sampling by taking two independent samples at each sampling event. One additional tubing volume of fluid will be purged from the well between collection of independent samples. Field duplicate samples will be taken at a frequency of one for each ten or fewer investigative samples of the same matrix. For this project, one duplicate ground water sample will be taken during each quarterly sampling as a quality assurance measure.

9. DOCUMENTATION OF FIELD SAMPLING ACTIVITIES

Field sampling activities will be documented following the procedures from the WMIMGS detailed in Appendix A-6, Item 4.2.2.2, on a Field Information Form also presented in that appendix. Analyses to be taken are documented on the Field Chain of Custody Form, previously presented in Appendix A-5. Additionally, field activities will be documented in a bound field log book as specified in the GWMP Section 7.4.2.8.

10. SUMMARY OF SAMPLING AND ANALYSIS

Tables VI-1 and VI-2 indicated the sampling and analysis to be performed.

VII. SAMPLE CUSTODY PROCEDURES

1. CHAIN OF CUSTODY PROCEDURES FOR FIELD ACTIVITIES

Chain of custody procedures for field activities are documented in Appendix A-5, Items 4.2.1.3, 4.2.1.4 and 4.2.2.1, from the WMIMGS. These items document the procedures which will be followed in receiving sample bottles and shipping containers from the analytical laboratory, maintaining sample integrity in the field and returning the filled sample bottles to the laboratory.

2. CHAIN OF CUSTODY PROCEDURES FOR LABORATORY ACTIVITIES

WMI Environmental Monitoring Laboratories will provide the sample bottles, AquaPaks, field chain of custody and field information forms for each sampling event. Once the samples arrive at the analytical laboratory (Enseco, Inc.), the Enseco chain of custody form and procedures for interlaboratory transfers will be used. Appendix A-7, an excerpt from the Enseco Quality Assurance Program Plan For Environmental Chemical Monitoring, (EQAPP), details the procedures to be followed.

3. FINAL EVIDENCE FILE

The final evidence file will reside with WMI Environmental Monitoring Laboratories (WMI EML). The final evidence file will consist of: all chain of custody reports from field and laboratory activities; field information forms; report from Enseco containing measured parameter values, internal QC check data, notes on any procedural abnormalities

occurring during analysis; report from WMI EML restating Enseco reported data and containing additional interpretation of data, correspondence pertaining to the project from the initiation of the drilling phase onward.

VIII. CALIBRATION PROCEDURES AND FREQUENCY

1. FIELD INSTRUMENTS

Chemical Waste Management will insure the accuracy of field measurements for conductivity, pH, temperature and dissolved oxygen through thorough pre-measurement calibration of the field instrumentation, and post-measurement verification. Manufacturer's recommendations on instrument calibration will be followed. Field instruments will be calibrated according to the general procedures and at the frequency indicated in Appendix A-8, an excerpt from the WMIMGS.

Field measurements of conductivity, pH, temperature and dissolved oxygen during well purging and sampling will be performed through the usage of a flow cell. A representative split of the fluids pumped from the well will be directed through the flow cell by means of a diverter valve to reduce instrument degradation due to excessive volumes of possibly abrasive fluid passing the sensors.

The calibrations will be checked every four hours while well purging is underway, and at the beginning and end of sample collection. This calibration check will consist of diverting the purge stream from the flow cell, removing the instrument probes and rinsing them thoroughly with deionized water, making four replicate measurements of a single known standard, preferable at a value relatively close to that being monitored in the purge or sample stream.

Instrument re-calibration will be required if: the mean of the four replicate pH measurements varies by greater than ±0.2 pH units of the standard; the mean of four replicate specific conductivity measurements varies by more than ±10% of the standard; the mean of four replicate temperature measurements varies by more than ±1.0°C of the standard; the mean of four replicate dissolved oxygen measurements varies by more than ±5.0% of the standard.

2. LABORATORY INSTRUMENTS

General calibration procedures and frequency for major laboratory instruments are discussed in Appendix A-9 from the EQAPP. More specific additional data on the calibration of instrumentation is found within the individual Standard Operating Procedures (SOPs) in Section IX of this QAPjP, and its referenced appendices.

IX. ANALYTICAL PROCEDURES

Specific Standard Operating Procedures (SOPs) to be utilized in evaluating required parameters are referenced from the following sections. Method reporting limits and the applicable dynamic or linear range of the method is specified in the SOPs. QAPjP Appendix A-10 contains tables listing laboratory reporting limits (analogous to the EPA's PQL) for the project and information on the effects any required sample dilution may have on these limits. Laboratory analytical SOPs are found in Appendix B of this QAPjP.

1. TAM PACKER PARAMETER LIST

PARAMETER	ENSECO METHOD	SOP's APPENDIX LOCATION
Ethylbenzene	LM-RMA-3001 (VOA by GC/MS)	B-13
1,2-Dichloroethane	"	"
Toluene	"	"
Chlorobenzene	"	"
Tetrachloroethene	"	"
1,2-Dichloroethene (Total)	"	"
trans-1,3-Dichloro-propene	"	"
cis-1,3-Dichloropropene	"	"
Carbon Tetrachloride	"	"
Chloroform	"	"
1,1,1-Trichloroethane	"	"
Bromomethane	"	"
Chloromethane	"	"
Chloroethane	"	"
Vinyl Chloride	"	"
Methylene Chloride	"	"
Bromoform	"	"
Bromodichloromethane	"	"
1,1-Dichloroethane	"	"
1,1-Dichloroethene	"	"
1,2-Dichloropropane	"	"

PARAMETER	ENSECO METHOD	SOP's APPENDIX LOCATION
1,1,2-Trichloroethane	"	"
Tricloroethene	"	"
1,1,2,2-Tetrachloroethane	"	"
Methanol	LM-ERC-3004	B-18
Methyl Ethyl Ketone	LM-RMA-3001 (VOA by GC/MS)	B-13
Total Phenols	LM-RMA-1099 (Total Recoverable Phenolics)	B-8
PCB's	LM-RMA-4003 (EPA 608/8080)	B-11
Dissolved Iron	LM-RMA-2037 (ICP)	B-3
Dissolved Nickel	"	"
Dissolved Arsenic	LM-RMA-2006 (GFAA)	B-4
Dissolved Cadmium	LM-RMA-2037 (ICP)	B-3
Dissolved Chromium	"	"
Dissolved Lead	LM-RMA-2006 (GFAA)	B-4
Dissolved Zinc	LM-RMA-2037 (ICP)	B-3
Dissolved Barium	"	"
Dissolved Mercury	LM-RMA-2005 (CVAAS)	B-5
Dissolved Selenium	LM-RMA-2006 (GFAA)	B-4
Dissolved Silver	LM-RMA-2037 (ICP)	B-3
Fluoride	LM-RMA-1025 (Anions by IC)	B-6
Calcium	LM-RMA-2037 (ICP)	B-3
Magnesium	"	"
Manganese	"	"
Potassium	"	"
Sodium	"	"
Boron	"	"
Bromide	LM-RMA-1025 (Anions by IC)	B-6
Chlorides	"	"
Nitrate	"	"
Nitrogen Ammonia	LM-RMA-1079	B-7
Sulfates	LM-RMA-1025 (Anions by IC)	B-6
Total Dissolved Carbonate	LM-RMA-1071 (Alkalinity section)	B-2
Total Dissolved Solids	LM-RMA-1060 (TDS)	B-1
Alkalinity	LM-RMA-1071 (Alkalinity section)	B-2
pH	LM-RMA-1071 (pH section)	B-2
Specific Gravity	LM-RMA-1101 (Hydrometric)	B-17
Specific Conductance	LM-RMA-1071 (SC section)	B-2
Temperature	Field Measured	NA

2. APPENDIX IX INCLUSIVE PARAMETER LIST

PARAMETER	ENSECO METHOD	SOP's APPENDIX LOCATION
Calcium	LM-RMA-2037 (ICP)	B-3
Magnesium	"	"
Manganese	"	"
Potassium	"	"
Sodium	"	"
Boron	"	"
Bromide	LM-RMA-1025 (Anions by IC)	B-6
Chlorides	"	"
Fluoride	"	"
Nitrate	"	"
Nitrogen Ammonia	LM-RMA-1079	B-7
Sulfate	LM-RMA-1025 (Anions by IC)	B-6
Total Dissolved Carbonate	LM-RMA-1071 (Alkalinity)	B-2
Specific Conductance	" (SC section)	"
pH	" (pH section)	"
Total Dissolved Solids	LM-RMA-1060 (TDS)	B-1
Specific Gravity	LM-RMA-1101 (Hydrometric)	B-17
Alkalinity	LM-RMA-1071 (Alkalinity)	B-2
Appendix IX Parameters From 40 CFR Part 264	See Following Pages	
Cyanide	LM-RMA-1018 (Method)	B-19
"	LM-RMA-1020 (Preparation)	B-19

APPENDIX IX PARAMETER	ENSECO METHOD	SOP's APPENDIX LOCATION
DIMETHOATE DISULFOTON FAMPHUR METHYL PARATHION PARATHION PHORATE THIONAZIN	LM-RMA-4006 (EPA 614/8140) Organophosphorous Pesticides	B-10
2,4,5-TRICHLOROPHENOXYACETIC ACID 2,4-DICHLOROPHENOXYACETIC ACID 2-(2,4,5-TRICHLOROPHENOXY)PROPIONIC ACID	LM-RMA-4007 (EPA 615/8150) Phenoxyacid Herbicides	B-9

APPENDIX IX PARAMETER	ENSECO METHOD	SOP's APPENDIX LOCATION
1,1,1,2-TETRACHLOROETHANE		
1,1,1-TRICHLOROETHANE		
1,1,2,2-TETRACHLOROETHANE		
1,1,2-TRICHLOROETHANE		
1,1-DICHLOROETHANE		
1,1-DICHLOROETHENE		
1,2,3-TRICHLOROPROPANE		
1,2-DIBROMO-3-CHLOROPROPANE		
1,2-DIBROMOETHANE		
1,2-DICHLOROETHANE		
1,2-DICHLOROPROPANE		
1,4-DICHLORO-2-BUTENE(TOTAL)		
2-BUTANONE		
2-HEXANONE		
3-CHLOROPROPENE		
4-METHYL-2-PENTANONE	LM-RMA-3001 (VOA by GC/MS)	B-13
ACETONE		
BATCH SEQUENCE FILE NAME		
BENZENE		
BROMODICHLOROMETHANE		
BROMOFORM		
BROMOMETHANE		
CARBON DISULFIDE		
CARBON TETRACHLORIDE		
CHLOROBENZENE		
CHLOROETHANE		
CHLOROFORM		
CHLOROMETHANE		
Chloroprene		
CIS-1,3-DICHLOROPROPENE		
DIBROMOCHLOROMETHANE		
DIBROMOMETHANE		
DICHLORODIFLUOROMETHANE		
ETHYLBENZENE		
IODOMETHANE		
METHYLENE CHLORIDE		
PENTACHLOROETHANE		
STYRENE		
TETRACHLOROETHENE		
TOLUENE		
TRANS-1,2-DICHLOROETHENE		
TRANS-1,3-DICHLOROPROPENE		

APPENDIX IX PARAMETER	ENSECO METHOD	SOP's APPENDIX LOCATION
TRICHLOROETHENE		
TRICHLOROFLUOROMETHANE		
VINYL ACETATE		
VINYL CHLORIDE		
XYLENE(TOTAL)		
	LM-RMA-3001 (VOA by GC/MS)	B-13
ACETONITRILE		
ACROLEIN		
ACRYLONITRILE		
BATCH SEQUENCE FILE NAME		
ETHYL METHACRYLATE		
ISOBUTYL ALCOHOL		
METHACRYLONITRILE		
METHYL METHACRYLATE		
P-DIOXANE		
PROPIONITRILE		
PYRIDINE		

APPENDIX IX PARAMETER	ENSECO METHOD	SOP's APPENDIX LOCATION
1,2,4,5-TETRACHLOROBENZENE		
1,2,4-TRICHLOROBENZENE		
1,2-DICHLOROBENZENE		
1,3,5-TRINITROBENZENE		
1,3-DICHLOROBENZENE		
1,3-DINITROBENZENE		
1,4-DICHLOROBENZENE		
1,4-NAPHTHOQUINONE		
1-NAPTHYLAMINE		
2,3,4,6-TETRACHLOROPHENOL		
2,4,5-TRICHLOROPHENOL		
2,4,6-TRICHLOROPHENOL		
2,4-DICHLOROPHENOL		
2,4-DIMETHYLPHENOL		
2,4-DINITROPHENOL		
2,4-DINITROTOLUENE		
2,6-DICHLOROPHENOL		
2,6-DINITROTOLUENE		
2-ACETYLAMINOFLUORENE		
2-CHLORONAPHTHALENE		
2-CHLOROPHENOL		
2-METHYLNAPHTHALENE		
2-METHYLPHENOL		
2-NAPTHYLAMINE	LM-RMA-3013 (BNA-625)	B-12
2-NITROANILINE	Semivolatile Organics by GC/MS	
2-NITROPHENOL		
2-PICOLINE		
3,3'-DICHLOROBENZIDINE		
3,3'-DIMETHYLBENZIDINE		
3-METHYLCHOLANTHRENE		
3-METHYLPHENOL		
3-NITROANILINE		
4,6-DINITRO-2-METHYLPHENOL		
4-AMINOBIPHENYL		
4-BROMOPHENYL-PHENYL ETHER		
4-CHLORO-3-METHYLPHENOL		
4-CHLOROANILINE		
4-CHLOROPHENYL PHENYL ETHER		
4-METHYLPHENOL		
4-NITROANILINE		
4-NITROPHENOL		

APPENDIX IX PARAMETER	ENSECO METHOD	SOP's APPENDIX LOCATION
4-NITROQUINOLINE 1-OXIDE		
5-NITRO-O-TOLUIDINE		
7,12-DIMETHYLBENZ(A)ANTHRACENE		
ACENAPHTHENE		
ACENAPHTHYLENE		
ACETOPHENONE		
ALPHA, ALPHA-DIMETHYLPHENETHYLAMINE		
ANILINE		
ANTHRACENE		
ARAMITE		
BATCH SEQUENCE FILE NAME		
BENZ(A)ANTHRACENE		
BENZO(A)PYRENE		
BENZO(B)FLUORANTHENE		
BENZO(GHI)PERYLENE	LM-RMA-3013 (BNA-625)	B-12
BENZO(K)FLUORANTHENE	Semivolatile Organics by GC/MS	
BENZYL ALCOHOL		
BIS(2-CHLOROETHOXY)METHANE		
BIS(2-CHLOROETHYL)ETHER		
BIS(2-CHLOROISOPROPYL)ETHER		
BIS(2-ETHYLHEXYL)PHTHALATE		
BUTYLBENZYLPHTHALATE		
CHRYSENE		
DI-N-BUTYLPHTHALATE		
DI-N-OCTYLPHTHALATE		
DIALLATE		
DIBENZ(A,H)ANTHRACENE		
DIBENZOFURAN		
DIETHYLPHTHALATE		
DIMETHYLPHTHALATE		
DINOSEB		
DIPHENYLAMINE		
ETHYL METHANESULFONATE		
FLUORANTHENE		
FLUORENE		
HEXACHLOROBENZENE		
HEXACHLOROBUTADIENE		
HEXACHLOROCYCLOPENTADIEN		
HEXACHLOROETHANE		
HEXACHLOROPHENE		
HEXACHLOROPROPENE		

APPENDIX IX PARAMETER	ENSECO METHOD	SOP's APPENDIX LOCATION
INDENO(1,2,3-CD)PYRENE		
ISODRIN		
ISOPHORONE		
ISOSAFROLE		
METHAPYRILENE		
METHYL METHANESULFONATE		
N-NITROSODI-N-BUTYLAMINE		
N-NITROSODI-N-PROPYLAMINE		
N-NITROSODIETHYLAMINE		
N-NITROSODIMETHYLAMINE		
N-NITROSODIPHENYLAMINE		
N-NITROSOMETHYLETHYLAMINE		
N-NITROSOMORPHOLINE		
N-NITROSOPIPERIDINE	LM-RMA-3013 (BNA-625)	B-12
N-NITROSOPYRROLIDINE	Semivolatile Organics by GC/MS	
NAPTHALENE		
NITROBENZENE		
O,O,O-TRIETHYL PHOSPHOROTHIOATE		
O-TOLUIDINE		
P-(DIMETHYLAMINO)AZOBENZENE		
P-PHENYLENEDIAMINE		
PENTACHLOROBENZENE		
PENTACHLORONITROBENZENE		
PENTACHLOROPHENOL		
PHENACETIN		
PHENANTHRENE		
PHENOL		
PRONAMIDE		
PYRENE		
PYRIDINE		
SAFROLE		
SULFOTEPP		

APPENDIX IX PARAMETER	ENSECO METHOD	SOP's APPENDIX LOCATION
4,4'-DDD		
4,4'-DDE		
4,4'-DDT		
ALDRIN		
ALPHA-BHC		
AROCLOR 1016		
AROCLOR 1221		
AROCLOR 1232		
AROCLOR 1242		
AROCLOR 1248		
AROCLOR 1254		
AROCLOR 1260	LM-RMA-4003 (EPA 608/8080) Organochlorine Pesticides, PCB's	B-11
BETA-BHC		
CHLORDANE		
CHLOROBENZILATE		
DELTA-BHC		
DIELDRIN		
ENDOSULFAN I		
ENDOSULFAN II		
ENDOSULFAN SULFATE		
ENDRIN		
ENDRIN ALDEHYDE		
HEPTACHLOR		
HEPTACHLOR EPOXIDE		
KEPONE		
LINDANE (GAMMA-BHC)		
METHOXYCHLOR		
TOXAPHENE		

3. BRASSFIELD PARAMETER LIST

PARAMETER	ENSECO METHOD	SOP's APPENDIX LOCATION
pH	LM-RMA-1071 (pH section)	B-2
Specific gravity	LM-RMA-1101 (Hydrometric)	B-17
Total Organic Carbon (TOC)	LM-RMA-1066	B-15
Sulfate (SO_4)	LM-RMA-1025 (Anions by IC)	B-6
Acidity (as $CaCO_3$)	LM-RMA-1073	B-16
Total Solids @ 105 °C	LM-RMA-1100	B-20
Total Suspended Solids	LM_RMA-1062	B-14
Chlorides	LM-RMA-1025 (Anions by IC)	B-6
Dissolved Chromium	LM-RMA-2037 (ICP)	B-3
Dissolved Iron	"	"
Dissolved Nickel	"	"
Dissolved Zinc	"	"
Dissolved Lead	LM-RMA-2006 (GFAA)	B-4
Conductivity @ 25 °C	LM-RMA-1071 (SC section)	B-2

4. FIELD MEASUREMENTS

4.1 BEFORE WELL PURGING

Prior to purging and sampling of the wells, the vapors above the standing fluid level in the wells will be checked for possible explosion or toxicity danger by the use of a flame-ionization (FID) or photo-ionization (PID) meter. The meter used will be calibrated to manufacturer's specifications prior to use.

Use of either type meter in the field will consist simply of inserting the sampling

probe into the well head prior to measuring water level, purging or sampling, and monitoring the vapors contained within for a period of approximately one minute, or until the instrument reading stabilizes. If concentrations of organic vapors above 10,000 ppm (1%) are detected, the free vapor space in the well will be ventilated until the concentration falls below 10,000 ppm. (Note: 1% concentration in air is approximately the lower explosive limit [LEL] for hexane; 5% concentration is the approximate LEL for methane.) Measured values will be recorded in the field notebook.

4.2 DURING WELL PURGING AND SAMPLING

During purging and sampling of the well, conductivity, temperature, pH and dissolved oxygen will be monitored in the flow stream by the use of an in-line flow cell. The flow cell will accommodate multiple sensor probes which can be calibrated while in place within the flow cell. Following calibration of sensors according to manufacturer's recommendations, purging of the well will commence. Parameter values will be recorded versus produced fluid volume until at least three well volumes have been produced and parameter stabilization as defined in Section VI part 3.2.1 has occurred. Fluid sample collection will then be initiated, and field parameters will continue to be monitored until sampling is complete. Continuing calibration checks during purging and sampling will be carried out as described in QAPjP Section VIII part 1.

X. INTERNAL QUALITY CONTROL CHECKS

1. FIELD ACTIVITIES

1.1 CONTINUING CALIBRATION CHECKS

After initial calibration prior to field use, the instrumentation used for monitoring pH, conductivity, temperature and dissolved oxygen will be checked every four hours during use, re-calibrated if necessary, as detailed in Section XIII, Subsection 1. The instruments will also be calibrated at the completion of sampling.

1.2 REPLICATE ANALYSIS

Replicate samples (field duplicate samples) will be taken at a frequency of one for each ten or fewer investigative samples of the same matrix. For this project, one duplicate ground water sample will be taken during each quarterly sampling as a quality assurance measure.

As documented in Table III-1 and III-2, each time an environmental sample is taken (make up water, drilling mud, ground water), a replicate (duplicate) sample will also be collected and sent to the analytical laboratory. During the drilling, completion and initial sampling of the Knox-Kerbel well a total of 10 duplicate samples will be collected. During the drilling, completion and initial sampling of the Brassfield well a total of 4 duplicate samples will be collected.

The collection of independent environmental samples, duplicate sampling and the establishment of baseline chemistry for the Knox-Kerbel monitor zone was previously addressed in QAPjP Section VI part 8.3.

1.3 BLANKS

Chemical Waste Management plans to utilize trip blanks and field blanks to aid in assuring and evaluating the quality of data obtained through the field sampling program and laboratory analytical procedures. The number of trip blanks, field blanks and duplicate samples was previously presented in Tables III-1 and III-2.

Trip blanks, field blanks and duplicate control samples provide independent checks on field sampling procedures, such as sample handling and storage. It should be noted that ground water analytical data is incomplete unless the analytical performance data (e.g., accuracy, precision, detection, and quantitation limits) are reported along with each set of results.

1.3.1 Trip Blanks

The Chemical Waste Management GWMP provides for the routine collection and analysis of quality control trip blanks. Each time a group of bottles is prepared for use in the field (a sampling event), two 40 ml trip blank vials will be

included in each shipping container used for volatile organics samples. These vials are filled with deionized water by the laboratory. The bottles filled with the blank will be transported to the sampling location and returned to the laboratory in a manner identical to the handling procedure used for the environmental samples. These trip blanks will be subjected to the same analysis as the ground water. The trip blank samples are used to check for any cross-contamination through the vial's septa during sample shipment and storage. The concentration levels of any contaminants found in the trip blank should not be used to correct the ground-water data. The contaminant levels should be noted, and if the levels are within an order of magnitude when compared to the field sample results, re-sampling of the ground-water will be performed.

The results of the analysis of the blanks should not be used to correct the ground-water data. If contaminants are found in the blanks, the source of the contamination will be identified and corrective action, including re-sampling, will be initiated.

1.3.2 Field Blanks

Field blanks will be prepared in the field during the sampling event, and consist of filling one sample container with the deionized water being used in the field which has been routed through the sampling device, including the field filtering

apparatus. Field blanks will be collected at the frequency of one for each ten or fewer investigative samples. (See QAPjP Tables III-1 and III-2 or VI-1 and VI-2.)

1.4 CALIBRATION STANDARDS

Calibration standards used in the field will be obtained from the analytical laboratory designated to perform sample analysis, or from other commercial sources. Calibration standard supplied by instrument manufacturers will be used if it has not attained its expiration date. All standards will be identified by supplier, lot number, purity/concentration, receipt or preparation date and expiration date.

2. LABORATORY ANALYSIS

Specific components and control limits for internal quality control checks are described in Appendix A-11. Appendix C-1 contains a SOP for internal laboratory controls in addition to a general discussion concerning the quality control measures and procedures utilized by Enseco.

XI. DATA REDUCTION, VALIDATION AND REPORTING

1. DATA REDUCTION

1.1 FIELD DATA

Field data is entered into the WMI EML Laboratory Information System from the Field Information Form. These data are reviewed by another person for accuracy prior to acceptance into the system. When anomalies occur, the Sampling Coordinator returns the Field Information Form to the sampler for correction or clarification.

1.2 LABORATORY DATA

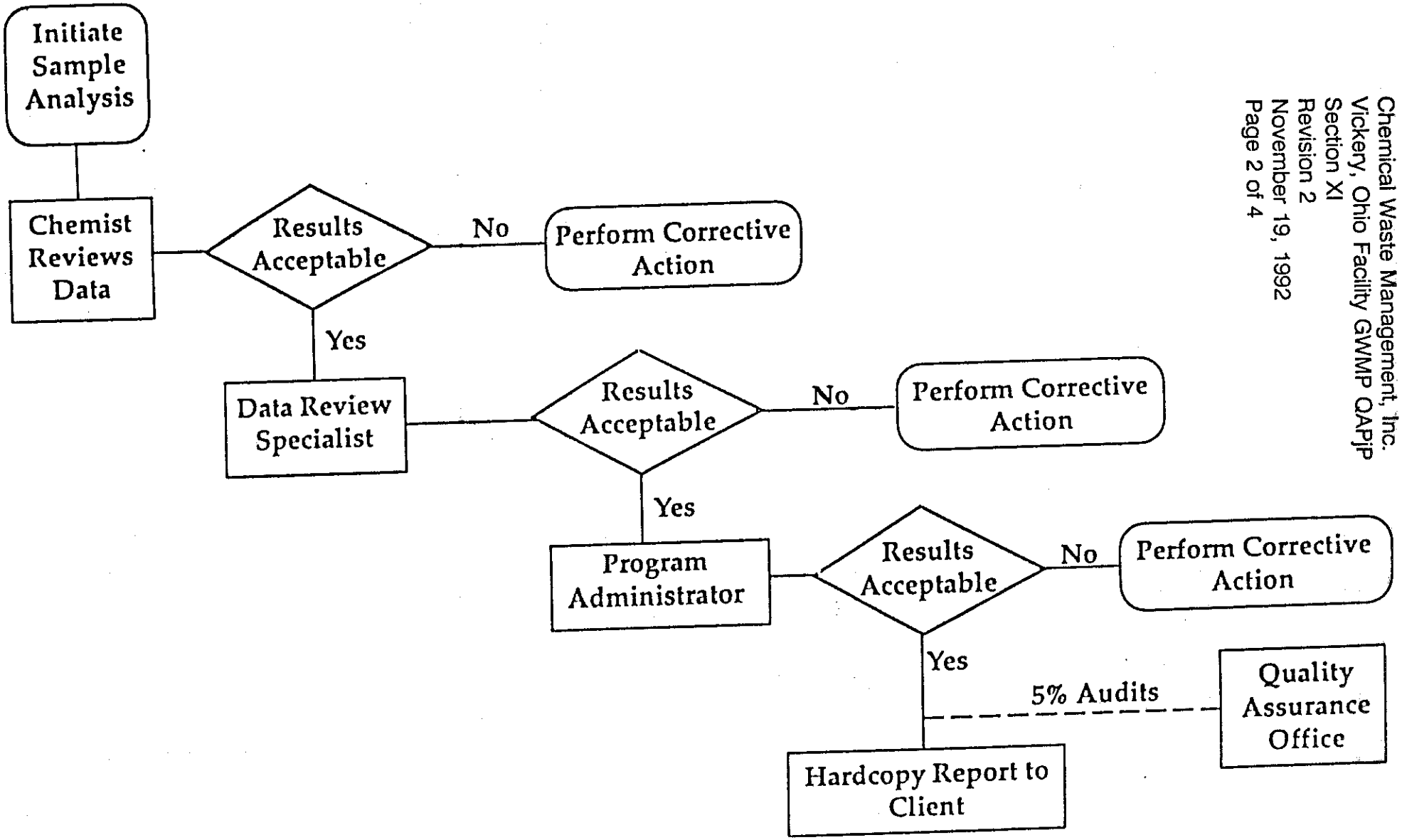
For specific information on Enseco's laboratory data reduction please refer to the individual SOPs. Laboratory guidelines for the reduction of data from raw form to reporting levels are documented in Appendix C-2, excerpted from the EQAPP.

2. DATA VALIDATION

All analytical data generated within the Enseco laboratory is subjected to extensive review to ensure validity prior to the generation of a report. Procedures and criteria utilized in Enseco's internal validation of data are described in Appendix C-2. Figure XI-1 demonstrates the internal data validation scheme.

FIGURE XI - 1

ENSECO INTERNAL VALIDATION PROCEDURE



Analytical data generated during the installation of the Knox-Kerbel monitoring well through the initial sampling of the Knox-Kerbel, will be subjected to full external validation procedures as described in Appendix H. After the Knox-Kerbel has been sampled for four quarterly monitoring periods including the initial sampling, and with USEPA approval, full data validation will be reduced to 25 percent of the analyses performed and the remaining 75 percent of analyses will be subjected to forms validation only. The analyses to receive full validation will be randomly selected from the population of available analyses without replacement on an annual basis. This will ensure that after the full validation of all analyses for the initial year of sampling, each analytical method receives a full validation annually. Should a new or modified analytical method be introduced into the GWMP, that method will be fully validated upon its first usage, then become subject to annual validation.

3. DATA REPORTING

Tabular data reporting summaries from WMI-EML will be provided in a manner similar to the example presented in Appendix C-3. The tabular data package will consist of at least the following:

1. Cover memo
2. Data Qualifier Report Information and Code Sheet
   (in Appendix II to Appendix C-3)
3. Data Qualifier Report
4. Event Summary Report
   (in Appendix III to Appendix C-3)
5. Client Report
   (in Appendix V to Appendix C-3)

6. Copies of chain of custody's and field forms
7. Copies of subcontract chain of custody's, if applicable

Raw data generated by and reported directly from Enseco, is to be utilized in the data validation procedures. An example of the Enseco data package is provided in Appendix G.

Additional data on report content and format was presented in Section 10 of the GWMP.

XII. PERFORMANCE AND SYSTEM AUDITS

Performance and systems audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in both the GWMP and in this QAPjP. Systems audits are conducted to verify that the necessary facilities, equipment, personnel and procedures are in place to ensure the generation of acceptable data. Performance audits are conducted to verify the ability to correctly identify or quantify parameters in check samples submitted by an auditing organization. Audits of field and laboratory procedures include both internal and external segments.

1. FIELD AUDITS

1.1 INTERNAL AUDITS

Party responsible for audits:	M. Grazioli WMI EML Quality Programs Coordinator II
Frequency of audits:	Annually
Methods and Procedures:	WMI EML 17 page audit procedure (included as QAPjP Appendix F) with an oral closing and a written report involving the sampling team.

1.2 EXTERNAL AUDITS

The USEPA Region V Central Regional Laboratory (CRL) and/or Central District Office (CDO) are responsible for external performance and system audits.

2. LABORATORY AUDITS

2.1 INTERNAL AUDITS

Enseco regularly conducts the following internal audits:

- Quarterly systems audits conducted by the Divisional Quality Assurance Officer.
- Periodic (at least yearly) audits conducted by the Corporate Quality Assurance Office.
- Special audits conducted by the Divisional Quality Assurance Director or Corporate Quality Assurance Office when a specific analytical problem is detected.

Additional data on internal performance and system audits is located in QAPjP Appendix A-12.

2.2 EXTERNAL AUDITS

The USEPA Region V Central Regional Laboratory (CRL) is responsible for external performance and systems audits.

USEPA Region V recently conducted an external audit of Enseco-RMA. Copies of various letters and memos from USEPA relevant to this audit are included in QAPjP Appendix A-12. The audit resulted in a finding that Enseco-RMA was acceptable for Appendix IX parameter analysis, with the implementation of various procedural changes recommended by USEPA CRL. The recommended changes have been implemented.

XIII. PREVENTATIVE MAINTENANCE

To minimize downtime and interruption of both field and laboratory work, preventive maintenance is routinely performed on each piece of analytical equipment.

1. FIELD EQUIPMENT

All field analytical equipment is checked for proper function before being taken to the work site. The equipment is calibrated before field use, checked for proper operation every four hours during use, and re-calibrated after usage. Equipment is thoroughly cleaned and tested prior to being returned to storage.

Any piece of equipment which does not function properly, or which can not attain satisfactory accuracy and precision results as indicated in Section VIII of this QAPjP will be serviced by trained personnel as required, or replaced if necessary.

2. LABORATORY EQUIPMENT

Designated laboratory personnel are trained in routine maintenance procedures for all major equipment. When repairs are necessary they are performed either by trained staff or trained service engineers employe by the instrument manufacturer.

Each laboratory has detailed SOPs on file that describe preventive

maintenance procedures and schedules. The laboratory also maintains detailed logbooks documenting the preventive maintenance and repairs performed on each analytical instrument.

The preventive maintenance schedule for major laboratory analytical equipment is documented in Appendix D.

XIV. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

The routine procedures used to assess data precision, accuracy and completeness are documented in Appendix E.

XV. CORRECTIVE ACTION

1. SUMMARY

Corrective actions may be required for two classes of problems: analytical and equipment problems and procedural noncompliance problems. Analytical and equipment problems may occur during sampling and sample handling, sample preparation, laboratory instrumental analysis, and data review. Procedural noncompliance problems may occur at any time during the project, from well installation and sampling through laboratory analysis and data reporting.

Any noncompliance with the established quality control procedures in the GWMP or QAPjP will be identified and corrected in accordance with the appropriate approved procedure. For noncompliance problems, a corrective action program will be determined and implemented at the time the problem is identified.

The person who identifies a problem is responsible for notifying his/her appropriate superior, and initiating a corrective action if appropriate. No staff member will initiate corrective actions without the prior communication of findings through appropriate channels.

For significant or recurring problems, the chain-of-command upward will ultimately assure that the CWM Project Manager is apprised of the situation. For significant or

recurring problems, the CWM Project Manager will notify the USEPA Region V Project Coordinator and OEPA Quality Assurance Officer concerning field related problems, and the Region V Project Coordinator, Quality Assurance Section and OEPA Quality Assurance Officer in the case of laboratory problems.

2. FIELD MEASUREMENTS AND SAMPLING

Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformances or suspected deficiencies of any activity or issued document by reporting the situation to the Site Manager or designee. During well installation and initial sampling, the Texas World Operations Site Manager (with acquiescence of the Texas World Project Manager) will have overall authority at the well site. During subsequent sampling events, the CWM Project Manager will designate a member of the sampling team as Site Manager. This manager will be responsible for assessing the suspected problems in consultation with the CWM Project Manager on making a decision based on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the Site Manager.

The Site Manager will be responsible for ensuring that corrective action for nonconformances are initiated by:

- evaluating all reported nonconformance;

- controlling additional work on nonconforming items;
- determining disposition or action to be taken;
- maintaining a log of nonconformance;
- reviewing nonconformance reports and corrective actions taken;
- ensuring nonconformance reports are included in the final facility documentation in project files.

If appropriate, the CWM Project Manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

Corrective action for field measurements may include:

- Repeat the measurement to check the error;
- Check for all proper adjustments for ambient conditions such as temperature;
- Check the proper operation of instrumentation;
- Check the instrument calibration;
- Replace the instrument or measurement devices;
- Stop work (if necessary).

The CWM Project Manager or his designee is responsible for all facility activities.

The Site Manager may at times find it desirable or necessary to adjust the facility's procedural programs to accommodate specific needs or conditions. When it becomes necessary to modify a procedure, the Site Manager notifies the CWM Project Manager (and Texas World Project Manager during the well installation / initial sampling). The CWM Project Manager clears any procedural changes with the USEPA Project Coordinator and OEPA Quality Assurance Officer. The Site Manager implements the necessary changes after obtaining approval from the CWM Project Manager.

Any change in the program procedures will be documented in the field log book as a field change request (FCR), that will be signed by the request initiators, Site Manager and CWM Project Manager. A copy of the FCR from the log book shall be attached to the file copy of the affected document (GWMP or QAPjP). The USEPA Project Coordinator and OEPA Qauality Assurance Officer must approve the change in writing or verbally prior to field implementation, if feasible. If unacceptable, the action taken during the period of deviation will be evaluated in order to determine the significance of any departure from established program procedures.

The CWM Project Manager is responsible for the controlling, tracking, and implementation of the identified changes. Reports on all changes will be distributed by the CWM Project Manager (or his designee) to all affected parties, which includes the USEPA Project Coordinator and OEPA Quality Assurance Oficer.

3. LABORATORY ANALYSES

When errors, deficiencies or out-of-control situations exist, the quality assurance plan provides systematic corrective procedures to resolve the problems and restore the proper functioning to the analytical system.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes and instrument sensitivity. If the problem persists or can not be identified, the matter is referred to the laboratory supervisor, manager and/or Quality Assurance Department for further investigation. Once resolved, full documentation of the corrective action is filed with the project records.

Specific corrective action procedures for laboratory analyses is provided in QAPjP Appendix A-13. Previously presented QAPjP Figure XI-1 indicated the points during analysis, data evaluation and validation at which corrective action may be performed.

XVI. QUALITY ASSURANCE REPORTS TO MANAGEMENT

1. FIELD OPERATIONS

During the drilling and installation of the monitor wells daily reports of progress will be provided to appropriate regulatory agencies (as indicated in Section 10 of the GWMP) and corporate management. A completion report will be filed documenting the procedures used during installation and completion, a discussion of any problems or unplanned occurrences during the process, and a description of corrective actions taken.

When quarterly, semi-annual or periodic reports are filed (described in GWMP Section 10), they will contain detailed information on sampling activities. Any abnormal occurrences will be noted, and corrective actions taken discussed.

2. ANALYTICAL LABORATORY

Project specific quality assurance data generated in conjunction with carrying out the required analyses will be included with the routine reports provided to the client, and in turn to regulatory agencies, to assist in evaluating the validity of the data generated. A standard data package as indicated by the example in QAPjP Appendix C-3 is planned for submission with each batch of analytical results.

Additional quality assurance data related to the project is retained by the laboratory and is available to regulatory agencies for external auditing purposes. This additional data includes, but is not limited to, such items as instrument calibration standard concentrations, surrogate and matrix spike solution concentrations and preparation methods, continuing calibration check standards, instrument printouts and strip charts, and corrective action reports relative to the project.

XVI. QUALITY ASSURANCE REPORTS TO MANAGEMENT

1. FIELD OPERATIONS

During the drilling and installation of the monitor wells daily reports of progress will be provided to appropriate regulatory agencies (as indicated in Section 10 of the GWMP) and corporate management. A completion report will be filed documenting the procedures used during installation and completion, a discussion of any problems or unplanned occurrences during the process, and a description of corrective actions taken.

When quarterly, semi-annual or periodic reports are filed (described in GWMP Section 10), they will contain detailed information on sampling activities. Any abnormal occurrences will be noted, and corrective actions taken discussed.

2. ANALYTICAL LABORATORY

Project specific quality assurance data generated in conjunction with carrying out the required analyses will be included with the routine reports provided to the client, and in turn to regulatory agencies, to assist in evaluating the validity of the data generated. A data package, as indicated by the examples in QAPjP Appendices C-3 and G, is planned for submission with each batch of analytical results. Additional quality assurance data related to the project and retained by the laboratory is available to regulatory agencies for external auditing purposes.

Reports concerning the independent validation of results from laboratory procedures will be prepared by the validating organization for each sampling event. Criteria for the validation procedures are provided in Appendix H.

The USEPA Region V Project Coordinator shall receive all QA reports.

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-3
SOP Number: LM-RMA-2037
Title: Metals Analysis: Inductively Coupled Plasma

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to EPA letters dated March 17, 1992 and September 15, 1992 to CWM containing comments (with additional attachments) regarding the CWM-Vickery GWMP QAPjP.

Section 8.1: Sample preparation SOPs LM-RMA-2002 and LM-RMA-2029 referenced from this section are located in QAPjP Appendix B-4A.

Section 8.4.1: The concentration of metals in the DCS QC samples will be documented in the project reports. Concentration of metals in the calibration standards will be available in the raw data. (Also see Section 6 of this SOP and the calibration concentration listing following this addendum.)

Section 9.2.9.1: Matrix spike levels and acceptance control limits are provided in QAPjP Appendix A-11.

SOP Appendix 1: Re: Arsenic, Selenium and Thallium. Enseco method detection limits and reporting limits as stated as stated in this appendix are correct, but are high relative to typical DQOs. However by reference to QAPjP Section IX it can be seen that these analytes will be evaluated by GFAA methods, and each has a reporting limit of 0.005 mg/l as per QAPjP Appendix A-10.

ICP Interelement Interference: Information on interelement intereference, noted in Section 3.3.2 of the SOP, has been added to QAPjP Appendix B-3. IEC composition is subject to change by lot number. Should a different lot number be in use at the time of analysis, additional information concerning the the composition and concentration of the standard solution used for interelement correction will be provided with the data report. (Also see Sections 3.1 and 6 of this SOP.)

ERA ICP

Calibration

Lot-XXXX

Standard # 2

Parameter	Concentration (in Standard mg/l)
Ag	1.00
Tl	1.00

ERA ICP

Calibration

Lot-XXXX

Standard # 4

Parameter	Concentration (in Standard mg/l)
Ca	100.
Li	100.
Mg	100.
K	100.
Si	100.
Na	100.

ERA ICP
Calibration
Lot - XXXX
Standard # 3

Parameter	Concentration (mg/L)
Sb	1.00
As	1.00
Ba	1.00
Be	1.00
Bi	1.00
B	1.00
Cd	1.00
Cr	1.00
Co	1.00
Cu	1.00
Pb	1.00
Mn	1.00
Mo	1.00
Ni	1.00
P	1.00
Sr	1.00
Sn	1.00
Ti	1.00
V	1.00
Zn	1.00
Al	10.0
Fe	10.0
Li	10.0
Se	10.0
Ca	10.0
Mg	10.0
Na	10.0

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-4
SOP Number: LM-RMA-2006
Title: Metals: GFAA Analysis

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to EPA letters dated March 17, 1992 and September 15, 1992 to CWM containing comments (with additional attachments) regarding the CWM-Vickery GWMP QAPjP.

Dynamic Linear Range:	The dynamic linear range for each analyte is addressed in Attachment 1 of the SOP, page 13 of 15. For this project As, Se, Pb, Tl, Cr, and Sb will be analyzed by GFAA. The standard concentrations for each of these elements are 0, 20, 50 and 100 ug/L and the linear range is 0 to 100 ug/L.
Section 7.:	Concentrations of the stock standard solutions and working standard solutions will be indicated in the reporting package.
Section 9.1.4:	Duplicate analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.
Section 9.1.5:	Matrix spike analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.
Section 9.2:	SW-846 methods, on which many Enseco methods are based, do not contain control limits for matrix spike and replicate analysis. The SW-846 methods do however contain limits on calibration checks. These checks are ±20 percent or greater, thus indicating that the accuracy limit on a matrix spike can never be greater than 80 to 120 percent. Enseco believes that the control limits indicated in QAPjP Appendix A-11 are realistic, based on the information in the methods and internally developed quality assurance data.

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-5
SOP Number: LM-RMA-2005
Title: Mecury Preparation and Analysis

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to EPA letters dated March 17, 1992 and September 15, 1992 to CWM containing comments (with additional attachments) regarding the CWM-Vickery GWMP QAPjP.

Dynamic Range: Dynamic range is 0.0002 ppm to .002 ppm Hg concentration.

Section 7.: Preparation of laboratory control sample and calibration stock solutions are addressed in SOP LS-RMA-4501, now a part of QAPjP Appendix B-5.

Five levels of standard solutions are used for instrument calibration, as noted in Sections 9.1.1 and 9.1.2.

Section 9.1.2: Concentration of the five standard solutions used in calibration are 0.0 (a blank from Section 9.1.1), 0.0002, 0.0005, 0.001, and 0.002 ppm, based on the stock standard preparation method in LS-RMA-4501, and the calibration preparation methods in Section 9.1.2.

Section 9.5: Duplicate analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.

Matrix spike analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.

SW-846 methods, on which many Enseco methods are based, do not contain control limits for matrix spike and replicate analysis. The SW-846 methods do however contain limits on calibration checks. These checks are ±20 percent or greater, thus indicating that the accuracy limit on a matrix spike can never be greater than 80 to 120 percent.

Enseco believes that the control limits indicated in QAPjP Appendix A-11 are realistic, based on the information in the methods and internally developed quality assurance data.

Section 11.1: An example of the data reporting package is provided in QAPjP Appendices C-3 and G.

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-6
SOP Number: LM-RMA-1025
Title: Anion Analysis by Ion Chromatography

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to EPA letters dated March 17, 1992 and September 15, 1992 to CWM containing comments (with additional attachments) regarding the CWM-Vickery GWMP QAPjP.

Section 9.1.4: Duplicate analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.

Section 9.1.5: Matrix spike analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.

SW-846 methods, on which many Enseco methods are based, do not contain control limits for matrix spike and replicate analysis. The SW-846 methods do however contain limits on calibration checks. These checks are ±20 percent or greater, thus indicating that the accuracy limit on a matrix spike can never be greater than 80 to 120 percent. The 80 to 120 percent limits will be utilized.

Section 11.: An example of the data reporting package is provided in QAPjP Appendices C-3 and G.

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-7
SOP Number: LM-RMA-1079
Title: Ammonia Nitrate + Nitrite TRAACS

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to EPA letters dated March 17, 1992 and September 15, 1992 to CWM containing comments (with additional attachments) regarding the CWM-Vickery GWMP QAPjP.

Section 1.3: Sample distillation will not be used in this ammonia analysis. Distillation is only performed for NPDES projects.

Section 9.1.4: The spiking concentration is 1 mg/L as N after the dilution of 1 ml of Intermediate Standard to 10 ml total volume using the sample. This is due to the Intermediate Sample having a concentration of 10 mg/L of N, as indicated in the preparation described in Sections 7.3.1, 7.3.2, 7.3.3 and 7.3.4 of the SOP.

Section 9.2: Control limits for blanks are described in QAPjP Appendix C-1, section 4.2.3. Also, Enseco's laboratory policy is that the concentration of the target analytes in the blank must be below two times the reporting limit.

A copper/cadmium column will be used in the analysis for the determination of nitrate + nitrite. Calibration standards will be treated in the same way as the environmental samples.

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-8
SOP Number: LM-RMA-1099
Title: Total Recoverable Phenolics

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to EPA letters dated March 17, 1992 and September 15, 1992 to CWM containing comments (with additional attachments) regarding the CWM-Vickery GWMP QAPjP.

Section 9: Laboratory Control Samples are routinely used to access the precision and accuracy of analytical results, as detailed in QAPjP Appendix C-1.

Section 9.1.4: Duplicate analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.

Section 9.1.5: Matrix spike analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.

Section 9.2: SW-846 methods, on which many Enseco methods are based, do not contain control limits for matrix spike and replicate analysis. The SW-846 methods do however contain limits on calibration checks. These checks are ±20 percent or greater, thus indicating that the accuracy limit on a matrix spike can never be greater than 80 to 120 percent. The 80 to 120 percent limits will be utilized.

Section 11: An example data reporting package is presented in QAPjP Appendices C-3 and G.

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-9
SOP Number: LM-RMA-4007
Title: Phenoxyacid Herbicides
by Electron Capture Gas Chromatography

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to EPA letters dated March 17, 1992 and September 15, 1992 to CWM containing comments (with additional attachments) regarding the CWM-Vickery GWMP QAPjP.

Section 1.1: Target compounds are listed in this section. Parameter retention time is variable with instrument condition and may vary slightly from calibration to calibration. A parameter list with relative retention times is provided following this addendum. Retention times recorded for an actual calibration are found in QAPjP Appendix B-9A.

Section 1.3: Linear dynamic range of compounds is determined from the instrument calibration, and normally covers one order of magnitude of analyte concentration. Herbicide target compound linear ranges are presented in a table at the front of QAPjP Appendix B-9A.

Section 6.2: Enseco-RMAL performs GC-ECD analyses using two dissimilar columns in order to accurately identify and quantitate organic compounds. Both columns are individually calibrated and must conform to constraints of ±20% difference for all compounds. When a compound is detected on both columns, the quantitative result is generally chosen from the lower of the two determinations. The higher value can reflect contamination from co-eluters, so the result is generally biased high. Based on this convention, Enseco-RMAL cannot designate one column as the primary column. Since quantitative data is taken from both columns throughout an analysis, both columns are, in a sense, equal to each other during the analysis. Only in extenuating circumstances will deviation from this procedure be this procedure be allowed. In those instances, the scientist will use his/her judgement to make accurate determinations.

Section 13.: See QAPjP Appendices C-3 and G for an example of the data reporting package.

Standards Preparation:
QAPjP Appendix B9-B contains four SOPs for the preparation of various standards utilized for herbicides analyses.

Injection Report

Acquired on 29-SEP-1992 at 14:39

RMAL Chromatography System on DENCR3

Analyst Name : RBRUGGEMAN
Lims Id : 2uL INJECTED
Comment : HP5890 Dual ECD Columns/RTx-5,RTx-20,0.25um,30m,0.25mm id
Method Title : METHOD FOR HERBICIDES
Sample Name : HERB CSA 20% T=ST #6348 T=ST
Sample Id : #6348
Sample Type : Standard Amount=1.00000
Bottle No : 3

RB 10-6-92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
3	3.476	3.489	319568	853113	0.96722	Dalapon	2.1
12	9.516	9.524	263860	564065	0.47551	DCAA	2.1
13	9.738	9.742	178025	370899	0.09606	Dicamba	2.1
14	10.133	10.116	160091	465897	97.83182	MCPP	2.7
15	10.413	10.387	199801	632373	96.28028	MCPA	3.2
17	11.156	11.169	149696	407634	0.23563	Dichlorprop	2.9
18	11.582	11.600	261566	855374	0.46482	2,4-D	3.2
21	12.449	12.453	136257	371575		D-BOB	2.9
26	14.284	14.298	225902	830700	0.09664	Silvex	3.7
27	15.124	15.138	207490	837507	0.09633	2,4,5-t	3.7
29	17.111	17.124	202944	905671	0.96189	2,4-DB	4.5
30	17.244	17.253	252386	1193140	0.23927	Dinoseb	4.5

Totals			
Unknowns	179361	683624	N/A
Quantified	2557583	8287947	197.74547
Grand Total	2736944	8971571	197.74547

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... HERBUS1
Run sequence..... N0929
Calibration..... N0929
Internal standard calibration using area
Calibration last modified on 30-SEP-1992 at 11:25

Uncalibrated peaks use user factor (0.0000)

Injection Report

Acquired on 29-SEP-1992 at 14:39

RMAL Chromatography System on DENCR3

Analyst Name : RBRUGGEMAN
Lims Id : 2uL INJECTED
Comment : HP5890 Dual ECD Columns/RTx-5,RTx-20,0.25um,30m,0.25mm id
Method Title : METHOD FOR HERBICIDES
Sample Name : HERB CSA 20% T=ST #6348 T=ST
Sample Id : #6348
Sample Type : Standard Amount=1.00000
Bottle No : 3

RB 10/6/92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
2	4.169	4.173	56593	167072	0.90416	Dalapon	2.7
11	11.480	11.489	115408	334411	0.51688	DCAA	2.9
12	11.916	11.924	73934	201636	0.09651	Dicamba	2.9
13	12.218	12.191	67980	233689	103.61088	MCPP	3.5
14	12.929	12.889	82574	320814	99.16589	MCPA	3.7
15	13.911	13.920	58607	203503	0.23186	Dichlorprop	3.7
16	14.400	14.404	51762	166354		D-BOB	3.2
17	14.996	15.009	95831	435873	0.46725	2,4-D	4.0
21	18.391	18.400	80134	366029	0.09540	Silvex	4.5
22	20.253	20.267	66930	347748	0.09377	2,4,5-T	5.1
24	22.458	22.462	34567	204150	0.22287	Dinoseb	5.9
25	22.880	22.893	55229	328397	0.91993	2,4-DB	5.9

Totals			
Unknowns	77635	213577	N/A
Quantified	839550	3309676	206.32542
Grand Total	917184	3523253	206.32542

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... HERBUS1
Run sequence..... N0929
Calibration..... N0929
Internal standard calibration using area
Calibration last modified on 30-SEP-1992 at 11:26

Uncalibrated peaks use user factor (0.0000)

Add To B-9A

Herbicide Target Compound Linear Ranges

Instrument Levels

Compound	Low Point	High Point
Dalapon	0.100ug/mL	1.000ug/mL
DCAA	0.050ug/mL	0.500ug/mL
Dicamba	0.010ug/mL	0.100ug/mL
MCPP	10.00ug/mL	100.0ug/mL
MCPA	10.00ug/mL	100.0ug/mL
Dichloroprop	0.025ug/mL	0.250ug/mL
2,4-D	0.050ug/mL	0.500ug/mL
2,4,5-TP (Silvex)	0.010ug/mL	0.100ug/mL
2,4,5-T	0.010ug/mL	0.100ug/mL
2,4-DB	0.100ug/mL	1.000ug/mL
Dinoseb	0.025ug/mL	0.250ug/mL

4.3.7 Filtration

Regulatory guidelines and permit requirements for sample filtration in the field may vary from site to site. However, all samples (except those listed below) must be filtered through a 0.45 micron membrane pressure filter, unless regulatory requirements specify otherwise.

Samples Not Requiring Filtration	Exceptions - "Total" Samples Requiring Filtration[1]
*Alkalinity	*TOC
*Turbidity	*Total Hardness
*Total Suspended Solids (TSS)	*Total Kjeldahl Nitrogen
*Total Solids	*Total Cyanide
*Semi Volatile Organics	*Total Petroleum Hydrocarbons
*Volatile Organics (VOA)	*Total Phosphorus
*Total Organic Halides (TOX)	
*Total Metals	
*Any other analytes listed as Total refer to unfiltered samples (if it does not say Total, assume it is Dissolved) EXCEPT FOR THE ANALYTES APPEARING IN THE COLUMN TO THE RIGHT	
*Coliform	
*pH	
*Specific conductivity	
*Oil and grease	

[1] Note: These analytes require filtration whether listed as "Total" or not. For example, samples are filtered whether analyzed for "Total Cyanide" or "Cyanide".

Regulatory and permit requirements will generally specify whether to analyze, for example, for total mercury as opposed to mercury or dissolved mercury. Corporate policy is to filter all heavy metals samples unless otherwise specifically requested.

Site specific requirements are contained in each monitoring program for the sampling event. The requirements must be noted on the Field Chain-of-Custody Records in the column labeled "Filt. (Y/N)". Filtering is necessary to determine the ions and compounds that are dissolved in

solution in the groundwater (rather than those that are present as particulates.). Monitoring wells are not as fully developed as drinking-water wells and often contain silts and sediment that must be removed by filtration. If the water is not filtered, ions and compounds naturally present in, or absorbed on to, the suspended particles may be released when samples are preserved and analyzed. This release would result in much higher concentration levels than are actually are present as dissolved constituents in the groundwater.

Filtration and preservation of groundwater samples is an integral part of the monitoring program. Improper techniques during this process can destroy the integrity of the sample. All possible precautions should therefore be taken to ensure that no contamination sources are introduced during filtration or preservation.

Notes:

* Filtering should be performed immediately after or during (in line filters) sample collection and should be done in the field. When this is not possible, filtration should be completed as soon as possible after the sample has been collected (within 20 min.) and should be done under the most contaminant-free conditions available.

* Refer to the Well I.D. Chart for the range of pH and specific conductances, to assist in determining the order of filtering. Any sample suspected or known to have high contamination levels, as identified by the Program Manager or Regional and/or Site Engineer, is to be filtered last, to prevent any contamination of the clean samples through the use of the same filtering equipment.

* Filtering equipment must be dedicated for groundwater only. Do not use any filtering apparatus that is used for other procedures such as EP-TOX or TCLP.

* Surface water, private wells, and leachate samples are never filtered unless specifically required by permit

* Prefiltration bottles are <u>not</u> to be reused. The material and use of prefiltration bottles must be noted on the Field Chain-of-Custody Record.

4.3.7.1 Filtration Equipment and Procedures

The following list delineates the equipment and other requirements for proper filtering and preservation of groundwater samples when filtration is not performed in-line.

* Only where field filtration is not possible, use a clean bench space with a readily accessible sink. This should be isolated from possible contamination sources, including outside air and inside fumes;

* Stainless steel pressure filter holder (1.5 liter reservoir); (In some EPA Regions, stainless steel is expressly forbidden. Contact the appropriate Regional office for recommendations where this is true.)

* 0.45 um filters (142 mm. diameter);

* Nitrogen (OSHA Class D) tank and regulator;

* Glass funnels;

* Large supply of deionized or laboratory reagent quality water or reagent quality water;

* Miscellaneous supplies (e.g., paper towels, tools, markers);

* Statement of Work Form (S.O.W.) to ensure there is a proper bottle and preservative for each analysis or series of analyses required for that particular sample;

* Designation of which samples are to be filtered for each sample point; (See S.O.W.);

The following list is a step-by-step procedure for filtering and preserving a typical monitoring well sample when filtering is not performed in-line.

1. Make sure that all filtering apparatus has been completely cleaned with deionized or laboratory reagent quality water only and dried, and that all pressure fittings are tight.

2. Remove the top, or reservoir, portion of the filter holder and position a new 0.45 um (142 mm. diameter) filter on top of the filter support screen, making sure it is flush on all edges. Care should be taken in

handling the filter paper, which is easily torn. A small amount of deionized or reagent quality water may be used to wet the filter and hold it on the filter screen, when excessively windy field conditions exist.

3. Replace the top or reservoir portion of the filter holder, over the filter paper, and tighten with the three wing-type clamps (finger tight only with equal force).

4. Unscrew the insert cover and fill the reservoir with the sample to be filtered, using a clean, dry funnel.

5. Screw down the inlet cover and reservoir securely.

6. Use a new sample container.

7. Position the new bottle under the outlet valve, beneath the filter holder.

8. Open the nitrogen cylinder, regulate pressure to a maximum of 40 psi, and pressurize the reservoir. Use the minimal amount of air pressure necessary for filtration. The filtration is now in process.

9. When the sample bottle is full or the filtration flow ceases, turn the stopcock to the "off" position. Turn the pressure relief valve to the "on" position to release the pressure.

10. Check the Field Chain-of-Custody Record for a list of appropriate preservatives, add the proper preservative (which is provided next to the sample bottle in the bottle holder) to the filtered sample and recap the bottle. Invert the bottle several times to mix the sample. Refer to the Appendix (A13-A16), for specific procedures on verifying that sufficient preservatives were added.

11. Record the necessary information of the Field Information Form and Field Chain-of-Custody Record after every filter change.

12. Between samples, disassemble the filter holder and its components and thoroughly rinse (a minimum of three times) with deionized or laboratory reagent quality water. Air dry the filter holder completely or dry with white paper towels.

13. Reassemble the filter apparatus and begin the process again.

It is imperative that the proper filtration and preservation techniques be strictly followed. Although at times the process may become tedious and may even seem trivial, it is essential that procedures are meticulously followed at all times. Failure to do so may invalidate all the precautions taken during purging, sampling, and transport, thus rendering the subsequent analysis useless. This precise care is necessary, since many of the analytes are measured in the part-per-billion range.

4.3.7.2 In-Line Filtration

When using an in-line filtration system (e.g., Well Wizard, Gelman, Millipore), a minimum of three pump cycles of water must be allowed to pass through the filter before obtaining a sample. These filters should only be used for filtering inorganic parameters, unless analytical data is available to support the conclusion that no sample contamination will occur.

4.3.4 Filling Sample Bottles

Sample bottles should be filled directly from the bailer or pump with a minimal amount of air contact. Volatile organics (VOA) and TOX bottles should be headspace-free (i.e., no air bubbles in the sample bottle). (See Appendix A4 and A5 for VOA sampling procedure.) All samples must be filtered unless specifically stated otherwise. Specific filtering instructions are included on the S.O.W. from the EML. Where in-line filtration is not available, prefiltration bottles may be used to collect the samples. This collection method is to ensure that no sediment will be introduced into the filtered sample which could cause possible analytical errors. [Prefiltration bottles must be obtained from the EML with the AquaPaks]. (See Appendix for filtration procedures for sampling points not having in-line filtration.) Plastic prefiltration containers should only be used for inorganic analytes. Any organic analytes to be filtered must be placed in glass prefiltration containers. All bottles or containers must be kept in a clean and locked storage area.

When filling the sample bottles, the following procedures and precautions are essential:

1. Bottle caps should be removed carefully so that the inside of the cap is not touched. Caps should never be put on the ground. Caps for VOA vials contain a teflon lined septum. The teflon side of the septum must be facing the sample to prevent contamination of the sample through the septum.

2. Field technicians should wear appropriate gloves.

3. AquaPaks should have cold ice packs in them before taking them into the field to aid in the cooling process or samples.

NOTE: UNDER NO CIRCUMSTANCES SHOULD BOTTLES (SAMPLE OR PREFILTRATION) OR CAPS NOT SUPPLIED BY THE EML OR THE SUBCONTRACT LAB BE USED FOR ANY SAMPLING!

4. The sample bottles should be filled with a minimal amount of air contact, and without allowing the sampling equipment or personnel to touch the inside of the bottles or bottle caps.

   Tubing or hoses from Well Wizards or pumps must not touch or be placed in the sample bottles.

5. Samples that are to be filtered and preserved, should be placed in prefiltration bottles and filled completely to allow for any loss of water from sediment during filtering.

   After filtering, sufficient space should be available in the sample bottles for the addition of required preservatives. The bottle caps should then be replaced tightly.

6. Samples for TOX and volatiles that are not to be filtered will have preservatives in the bottles when received. These bottles should be completely filled with the sample, with as little overflow as possible, and the bottle caps should be replaced tightly.

7. TOX and VOA vials must be filled so that they are headspace-free. These sample bottles, therefore, need to be slightly overfilled (water tension will maintain a convex water surface in the bottle). The caps for these bottles should be replaced gently, to eliminate any air bubbles in the sample. These bottles must then be checked by inverting them and tapping them sharply with a finger. If any air bubbles appear, open the bottle, add more water, and repeat this process until all air bubbles are gone. Do not empty the bottle and refill it. For additional information on filling VOA vials, see pages A4 and A5.

8. Air space should be left in coliform sample bottles, because these samples must be shaken before analysis.

9. All sample bottles after required filtering, filling, and preserving, must be placed in a refrigerator or AquaPak with ice until ready to be shipped. Samples must be shipped to the EML or the subcontract lab

the day they are collected. Due to the extremely short hold and extraction times involved with many of these methods, it is necessary to ship the same day as sampling occurs. Therefore, allow time at the end of the day to get the collected samples to the courier.

10. Never place VOA or TOX bottles in direct contact with ice packs because they may cause the sample to freeze and the bottle to break.

11. Sample bottles, caps, or septums that fall on the ground before filling should be thoroughly rinsed with sample water before being used. All circumstances regarding dropped caps or bottles, and their subsequent rinsing and use, <u>must</u> be noted on the Field Information Form.

4.2.1.3 The AquaPak and Sample Bottles

The sampler or sampling team is responsible for checking all sample bottles and AquaPaks when received and for preparing ice packs prior to the sampling event. This person must immediately notify the EML of any problems or questions. The AquaPaks may also be shipped to the subcontractor or to the sampling site, depending on the location requested when the monitoring program is created. If AquaPaks are shipped to the site, the subcontractor must communicate with those on the site to ensure the timely arrival of AquaPaks. The subcontractor must also communicate with site personnel to ensure access to the site before the sampling event so that equipment, AquaPaks, and supplies may be checked. If the AquaPaks are shipped to the subcontract lab, the recipients must arrange with site's Program Manager who will contact the EML to arrange for the delivery of the AquaPaks to the sampling team. (Note: It is important that AquaPaks be sorted in buildings that do not contain solvents, cleaners or gasoline or diesel fuels.)

Bottles from more than one lab may be used when sampling. The following are some examples:

* All sample bottles and AquaPaks from the EML.
* Sample bottles and AquaPaks from both the EML and the subcontract lab.
* Bottles supplied by a regulatory agency for split samples.

The EML provides AquaPaks, which are rotary molded, high-density, plastic insulated sample containers. Each AquaPak also contains bottles packed in molded polyethene. Ice packs are provided to thermally preserve samples for up to 40 hours. Chain-of-custody seals, preservatives and forms for documentation of the sampling event, are also provided within the AquaPak.

The EML will notify the sampling team of AquaPak delivery arrangements and of the correct contents of all AquaPaks. Before the sampling event, the team leader will receive information for the sampling program on the Statement of Work (see Figure 4.4).

Upon receipt of the AquaPak, the gray numbered seal must be examined. If it is not intact, contact the EML immediately for instructions. If the seal is unbroken, break the seal, open the AquaPak, and remove the plastic field

bag from inside. (Document the seal number on the Field Chain-of-Custody Record.)

This bag contains Field Chain-of-Custody Record and Field Information Forms, red temporary seals, return labels, a Federal Express ticket, and one gray numbered seal. The gray seal is used as the final seal when the AquaPaks are returned to the lab. The red, unnumbered seals are used to seal the AquaPak if it is left unattended.

Fill out the Field Chain-of-Custody Record (Figures 4.7 and 4.7.1) and Field Information Form (Figures 4.8 and 4.8.1) when initially opening the AquaPak (e.g. record the seal number). If the samples are returned directly to the EML, send the white (original) and yellow copies of both the Field Chain-of-Custody Record and the Field Information Form with the samples. If samples are returned directly to a subcontract lab, send the pink copy of the subcontract Chain-of-Custody Record to the EML with the AquaPak.

Note the arrangement of the bottles and ice packs inside the AquaPaks before removing them. Repack them in the same order when sampling is complete. AquaPaks are packed for economy of space and often, unless special attention is given, it is difficult to fit everything back in place.

An inventory of the bottles and their condition should be noted. The bottle list, analyses required, and preservation requirements are noted on the Field Chain-of-Custody Record in the "AquaPak Contents" section. Each sample bottle is labeled with its own bottle I.D. number, which refers to the lab group, method of filtering, and necessary preservatives. All bottles used for a particular sample point must have the same bottle set number, since samples from several sample points may be packed in the same AquaPak.

New, properly cleaned sample bottles are provided in each AquaPak. The type of bottle will vary depending on the analysis required. For samples requiring preservatives, premeasured amounts of preservatives are supplied in small vials and are placed next to the proper bottle in the bottle holder. These preservatives should be added to the sample bottles after filling. However, the preservatives for the VOA and TOX methods are placed in the bottle prior to shipment. Each bottle is labeled with a sticker to identify the preservatives that are required. Preservatives must not be put in bottles that do not require them. Verification that the appropriate preservatives have been included with the bottles and that the appropriate

bottles are available is imperative. A list of sample bottles (type and size) and preservatives required for each analysis is included in the Appendix.

Each AquaPak that includes bottles for volatile organic analyses, also includes a trip blank (40 ml. vial filled with reagent free water). When volatile organic analyses have been requested, the AquaPak will contain a trip blank regardless of whether a request has been placed for analysis of the trip blank. Prior to shipment, the EML checks each trip blank vial to ensure that it has no air bubbles. Occasionally, due to pressure and temperature changes, small bubbles will appear in the blank. Note the condition of the blanks as, well as any relevant information about the AquaPak, on the Chain-of-Custody Record.

For field blank analyses, bottles will be included within the AquaPak. The water the sampling team uses in the field should be used for field blanks.

The AquaPak also contains ice packs to maintain sample temperature at the level required by EPA methods. The sampling period must be planned to allow adequate time for freezing of the ice packs. Usually a 24-hour period is required to ensure that the ice pack will be frozen solid. After the AquaPak is checked in, the ice packs should be placed in the freezer, and the Field Chain-of-Custody Record is signed. The AquaPak is then closed and a red seal is placed through the chain of custody hole. The red temporary seals are to be used whenever the AquaPak is left unattended. The gray seal is to be used after sample collection when returning the AquaPak to the EML.

If any questions arise, or if there are any problems with the AquaPaks and their contents, call the EML before proceeding.

4.4 Sample Preservation, Storage, and Shipment

4.4.1 Sample Preservation

Samples are to be preserved immediately after filtering or immediately after sample collection (if samples are not filtered). TOX and volatile organic samples (VOA's), which can have no headspace (no air bubbles trapped in the sample), have preservatives included in the sample bottle. When filling them, do not allow the bottles to overflow any more than necessary to eliminate headspace.

Premeasured amounts of preservative reagents are supplied with the sample bottles EML sends . The preservatives are provided in small vials and are packed in the bottle holders next to the bottle requiring preservation. The preservatives should be added to the sample bottle after it has been filled. Bottles must not be overfilled, and they should be inverted (once capped) to mix the preservative with the sample. Refer to the Appendix for procedures for correct preservation. Bottle lids must not be placed on the ground or interchanged among sample bottles. Empty preservative vials should be returned in the AquaPak with the sample bottles.

A listing of preservatives, by analysis, is included in Appendix A-4, pp A13-A16, for reference only. Do not use a substitute for the chemical preservative supplied, because the reagents are batched, controlled, and supplied by the lab group that willconduct the analysis. If substitutions are

made from stockpiles at a site, this fact should be noted on the Field Chain-of-Custody Record. At no time should preservatives be kept for greater than six months.

4.4.1.1 Temperature Control

The sample should be cooled to 4°C from the time the sample is taken through analysis. Samples should be maintained in temperature regulated refrigerators, in coolers, or in AquaPaks containing frozen ice packs. Provisions must be made in advance for facilities to freeze the ice packs. Most solid waste sites have no such facilities so it is recommended to bring prefrozen coolers and/or dry ice to the site. The ice packs should be frozen solid prior to use. See A-30 through A-32 for additional details on temperature preservation procedures.

4.4.2 Sample Packing and Storage

4.4.2.1 Checking Sample Codes and Numbers

The sampler must record the sample designations in the appropriate spaces on the Field Chain-of-Custody Records and Field Information Forms. It is extremely important that the proper designations be recorded in the proper space on the form and that they be double-checked before sealing the AquaPak (See A.6 and A.7). Proper sample designations are included on the Statement of Work, which is sent to the site or the consultant sampling team, before the start of the sampling event.

In addition, the ENS numbers, when assigned, must be indicated on all paperwork from the subcontract lab.

ALL BOTTLE SETS AND CORRESPONDING CHAIN-OF-CUSTODY RECORDS AND FIELD INFORMATION FORMS MUST HAVE THE SAME SAMPLE POINT DESIGNATION NUMBER AND ENS NUMBERS.

All bottles filled from the same sample point at the same time must have identical sample designations and bottle set numbers (except field duplicates). Bottle set labels should be double-checked for consistency between those being sent to the subcontract lab and those being sent to the EML. Samples that are split with regulatory agencies should also be checked for consistent sample point designation numbers and for other methods of identification used by the agency.

4.4.2.2 Sample Packing

After the sample is collected and the preservatives are added, when applicable, the bottles are capped and placed in the AquaPak. The frozen ice packs are then placed into the AquaPak. The Field Chain-of-Custody Records and Field Information Forms must then be reviewed to ensure that they have been completed. (See A.6 and A.7) All paperwork is then placed in the plastic bag, which is then sealed and placed inside the AquaPak.

All bottles should be wiped clean with paper towels before placement in the AquaPak. The bottle sets and ice packs should be repacked in the same configuration as when received. It is essential that VOA vials and TOX bottles should never be placed directly on the ice packs. The AquaPak must be kept as clean as possible to minimize the potential for contamination. All bottle caps should be checked to ensure they are tight and will not become loose when inserted in the AquaPak. Do not tape the bottle caps! Labels should be taped only if they are loose, and this should be noted on the Field Information Form or Chain-of-Custody Record.

Containers and packing materials designed to prevent breakage and spills must be used for shipping. In AquaPaks, tight shock-resistant bottle holders are provided around each glass bottle. The AquaPaks must never be shipped without the ice packs or the bottle holders in place. After the AquaPak is repacked and locked, the gray numbered seal is placed through the outside latch and locked. Finally, a black poly strap should be placed through the center channel, and over the gray seal of the AquaPak.

There are three important reminders for packing the AquaPaks:

1. Glass containers should not be packed in contact with each other. Bottle holders should be used for glass bottles to protect them from potential breakage.

2. In very cold weather the ice packs should be unfrozen or slushy.

3. If the ice packs appear to be leaking, they should be sealed in a sealable plastic bag before being placed in the AquaPak.

4.4.2.3 Sample Storage

Samples should be stored at 4°C, in an enclosed cooler or darkened refrigerator where possible, before shipment to the lab for analysis. Samples should be shipped daily to the lab to ensure proper temperature

control and holding holding time requirements are met. A listing of recommended holding times is in the Appendix (A13-A16) for reference.

4.4.3 Sample Shipment

4.4.3.1 AquaPak Transportation Arrangements

A member of the sampling team must be appointed to arrange sample pickup and transportation to the lab. Friday shipment of samples to the subcontract lab should be avoided to ensure that holding times are not exceeded over a weekend. This is especially true for coliform analyses because few labs analyze samples on the weekend. The EML accepts AquaPaks for delivery six days per week. Delivery requested on Saturday must be noted specifically on the shipping/packing air bill for the courier.

AquaPaks should be returned via Federal Express, if possible. When contacting the courier for transport of a sample, specify the AquaPak's contents. Alert the courier to the potential problems of the samples freezing in the winter or ice packs melting in the summer, and note these potential problems on the shipping/packing label. The courier must take care to minimize exposure of the AquaPaks to temperature extremes. AquaPaks should be received at the lab within 24 to 44 hours of when the frozen ice packs were placed in the AquaPak. This is necessary for temperature preservation and to meet required holding times for some analytes. Time lags must be documented on the Field Chain-of-Custody Record. Use the preaddressed shipping label supplied with the AquaPak and remove all old shipping labels.

For coolers containing samples from subcontract labs, the EML generally makes the arrangements for the subcontract lab for container delivery. The sampling team is responsible for notifying them of the time for sample pickup.

STANDARD OPERATING PROCEDURE (S.O.P.)

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1. **Title**-Sample Bottle Acquisition and Preparation

2. **Principle Reference**-Not applicable

3. **Application**-The bottle preparation area helps to insure the quality of the WMI EML's data providing our clients/regions with clean and contaminant-free sample bottles.

4. **Summary of Procedures**-Clean/Contaminant-Free Bottles are critical to the effectiveness of the laboratories and overall operations. Sample bottles are purchased from reliable sources as precleaned bottles (volatile bottles) or prepared as follows:

   4.1 The Heinicke Model Pulsonic 6000 Washer is used to triple rinse new bottles and caps with hot tap water.

   4.2 Next bottles are thoroughly rinsed with hot D.I.-Water (18 megohm quality) from the laboratory's Reverse Osmosis/Deionization System (RODI).

   4.3 Following the rinse, the bottles are placed in a Hotpack Model 214300 Tru-Temp Oven at 68 degrees C until dry.

   4.4 After a cooling period in a contaminant-free area, all caps are placed back on the appropriate bottles. All sample bottles are used once then discarded.

5. **Apparatus/Equipment/Materials-**

   5.1 Washer-The bottles are washed in a Heinicke Model Pulsonic 6000 Washer.

   5.2 Dryer-Bottles are dried in a Hotpack Model 214300 Tru-Temp Oven.

   5.3 Sample Bottles

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      5.3.1 Amber glass 40ml w/septum closure-brand I-Chem 100 and 200 Series.

      5.3.2 Amber glass 125, 250, 500, and 1000ml with Teflon lined lids-brand QORPAK.

      5.3.3 High density polyethylene 125, 250, 500 and 1000ml-brand V.W.R./ Nalgene.

6. **Safety**-Safety glasses and steel-toe safety shoes must be worn. At all times, Good Laboratory Practices will be followed.

7. **Procedure**-The AquaPaks contain several different types of sample bottles. Even though they all go into one AquaPak, the cleaning procedures vary. These bottles are not prepared with other laboratory glassware; glass bottles and plastic bottles are washed and dried in separate batches because of cross-contamination possibilities.

7.1 Amber Glass Bottles 125, 250, 500 and 1000ml brand-QORPAK; High Density Polyethylene Plastic Bottles 125, 250, 500 and 1000ml brand V.W.R./Nalgene. New bottles are washed, rinsed and drained in Heinicke Model Pulsonic 6000 Washer (5.1) and dried in Hotpack Model 214300 Tru-Temp Oven (5.2).

7.1.1 Caps are removed from bottles.

7.1.2 Bottles are inverted in wire washing racks, caps are placed in wire racks with plastic side guards or plastic baskets and placed in the washer (see 5.1).

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7.1.3 Power switch is pressed to start fill cycle of washer. Temperature is preset at 68 degrees C. When water is up to temperature, press the start button. The washer cycles through three 2 minute rinse cycles using hot tap water, and one 2 minute rinse with hot D.I. water from the laboratory's pure-water system.

7.1.4 Bottles and caps are allowed to drain for one minute. They are then drained further by vigorously shaking the wire racks to remove excess water.

7.1.5 Drained bottles and caps in racks are placed in the drying oven.

7.1.6 Power switch is turned on. Temperature is preset at 68 degrees C. Bottles are left in the dryer until dry (60-90 min.) indicated by water drops not being visible in any bottle or cap.

7.1.7 Caps are replaced on the appropriate bottles and tighten (finger tight). Bottles are then placed on the storage shelves in the Log-Out area.

7.2 Amber glass vial 40ml w/Septum Closure brand I-Chem
New precleaned 100 Series I-Chem vials as well as new precleaned/sealed Series 200 I-Chem vials are used without washing.

7.2.1 New precleaned 100 Series vials are stored in the Log-Out area until needed.

7.2.1.1 Temperature blanks are made using new precleaned 100 Series vials. They are reused for subsequent events.

7.2.1.2 Temperature blanks are reused by rinsing the outside of the vial with hot tap water and left to air dry.

7.2.2 Precleaned/sealed 200 Series vials are stored in the VOA Water Prep Room where they are preserved and/or filled with RODI Water as needed.

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8. Documentation Requirements

8.1 I-Chem protocol for precleaned bottles are specified in Appendix A.

8.2 Report from Quarterly Quality Check of representative bottle types are sent to Client Services Manager and Sr. Quality Program Coordinator.

8.3 Affected clients will be informed of any contamination found during the quarterly bottle quality check.

9. Quality Assurance and Quality Control

9.1 Bottles are inspected when they are received at the EML. Only the bottle suppliers specified in (5.3) will be accepted.

9.2 Bottle suppliers were chosen originally on the basis of contamination screening of the bottle types they supply. Continuing quality checks are completed quarterly through the following process:

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9.2.1 Two sets of bottles are filled with Reverse Osmosis Deionized water. One set of bottles are used as they are received from the supplier. The other set uses bottles which have been washed as in (7.1).

9.2.2 Analytes as follows:

Nitrogen, Total KJELDAHL C
Nitrogen, Ammonia Colorime
Phosphorous, Total
Phenols, High Range Auto
Total Organic Carbon Dupl
Chloride, Automated Ferri
Silver By ICP-Total
Barium BY ICP-Total
Cadium, By ICP-Total
Chromium By ICP-Total
Mercury By Cold Vapor AA-
Arsenic By GFAA-Total
Lead By GFAA-Total
Selenium by GFAA-Total
SV Organochlor.Pest.DW509
Meth 624 Publ Cmpd List

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9.2.3 Quality specifications of precleaned bottles may be found in Appendix A.

10. References-None

11. Appendices-

11.1 I-Chem Research Cleaning Protocol

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12. **Approval**-The original copy of this document was signed by:

Author	4-8-92 Date
Manager, Client Services	Date
Manager, Quality Programs	Date
Assistant Director	Date
Senior Director	Date

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I-CHEM RESEARCH CLEANING PROTOCOLS™ FOR THE PREPARATION OF CONTAINERS

— Protocol A™ —

- Amber Glass Bottles
- Clear Glass Bottles
- Wide Mouth Amber Glass Jars
- Wide Mouth Clear Glass Jars*

** Also available cleaned according to Protocol B™ - see below*

Cleaning Protocol A™ specifications:

1. Laboratory Grade Detergent Wash and Rinse
2. Acid, Deionized Water, and Solvent Rinses
3. Oven Drying, Capping and Packing under quality controlled conditions

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Sample Containers Prepared According to Protocol A™ Are Recommended For Use In The Analysis Of:

Acidity/Alkalinity
BOD (Biological Oxygen Demand)
Chloride
COD (Chemical Oxygen Demand)
Color
Conductivity
Cyanide
Extractable Organics
Hardness
Mercury
Metals
Oil & Grease
pH
Phenols
Phosphate
Settleable Residue/Suspended Solids
Silica
Sulfate
Sulfide
Sulfite
TOC (Total Organic Carbon)
Turbidity

— Protocol B™ —

- 40 ml Borosilicate Clear and Amber Glass Vials
- 20 ml Polyethylene vials
- 40 ml and 20 ml Flint Glass Vials
- 8 oz. and 4 oz. Septum Bottles
- Wide Mouth Clear Glass Jars* and High Density Polyethylene Bottles (allow extra time for delivery)

** Wide Mouth Clear Glass Jars prepared according to Protocol B™ are supplied with white Teflon® lined polypropylene closures attached*

Cleaning Protocol B™ specifications:

1. Laboratory Grade Detergent Wash and Rinse
2. Multiple Deionized Water Rinses
3. Oven Drying, Capping and Packing under quality controlled conditions

Sample Containers Prepared According to Protocol B™ Are Recommended For Use In The Analysis Of:

COD (Chemical Oxygen Demand)
Nitrate-Nitrite
Purgeable (Volatile) Organics
TOC (Total Organic Carbon)
Trihalomethanes

— Protocol C™ —

- High Density Polyethylene Bottles*
- Polyethylene Cubitainers
- High Density Polyethylene Wide Mouth Jars
- Genuine Nalgene Products

** Also available cleaned according to Protocol B™ - see above*

Cleaning Protocol C™ specifications:

1. Laboratory Grade Detergent Wash and Rinse
2. Acid Rinse
3. Multiple Deionized Water Rinses
4. Air Drying, Capping and Packing under quality controlled conditions

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Sample Containers Prepared According to Protocol C™ Are Recommended For Use In The Analysis Of:

All analysis noted for Protocol A™ above except:
BOD (Biological Oxygen Demand); COD (Chemical Oxygen Demand); Extractable Organics; Phenols; Oil & Grease; TOC (Total Organic Carbon)

™ *Protocol is a trademark of* I-Chem Research.

EML RECOMMENDATION FOR SAMPLING AND PRESERVATION OF SAMPLES ACCORDING TO ANALYTE[1]

Measurement	Vol. Req.(ml.)	Container[2]	Preservative[3,4]	Holding Time[5]
100 Physical Properties				
Color	50	P	Cool, 4°C	48 hrs.
Conductance	100	P	Cool, 4°C	28 days
Hardness	50	P	HNO_3 to pH<2	6 mos.
Odor	200	G only	Cool, 4°C	24 hrs.
pH	25	P	Cool, 4°C	Analyze Promptly Upon Receipt
Residue				
Filterable(TSS)	500	P	Cool, 4°C	7 days
Nonfilterable (TDS)	500	P	Cool, 4°C	7 days
Total(TS)	500	P	Cool, 4°C	7 days
Volatile	100	P	Cool, 4°C	7 days
Settleable Matter	1000	P	Cool, 4°C	48 hrs.
Temperature	1000	P	Cool, 4°C	Analyze Immediately
Turbidity	100	G	Cool, 4°C	48 hrs.
200 Metals				
Dissolved	500	P	Filter on site HNO_3 to pH<2	6 mos.
Suspended	200	P	Filter on site	6 mos.[8]

Measurement	Vol. Req.(ml.)	Container[2]	Preservative[3,4]	Holding Time[5]
Total	500	P	HNO_3 to pH<2	6 mos.
Chromium[6]	200	P	Cool, 4°C	24 hrs.
Mercury				
Dissolved	100	P	Filter HNO_3 to pH<2	28 days
Total	100	P	HNO_3 to pH<2	28 days
300 Inorganics, Non-Metallics				
Acidity	100	P	Cool, 4°C	14 days
Alkalinity	100	P	Cool, 4°C	14 days
Bromide	100	P	None Req.	28 days
Chloride	50	P	None Req.	28 days
Cyanides	500	P	Cool, 4°C NaOH to pH>12 0.6g ascorbic acid[6]	14 days[7]
Fluoride	100	P	None Req.	28 days
Nitrogen				
Ammonia	100	G	Cool, 4°C H_2SO_4 to pH<2	28 days
Kjeldahl, total	100	G	Cool, 4°C H_2SO_4 to pH<2	28 days
Nitrate plus Nitrite	100	P,G	Cool, 4°C	28 days
Nitrate plus Nitrite	100	P,G	Cool, 4°C H_2SO_4 to pH<2	28 days*
Nitrite	100	P,G	Cool, 4°C	48 hrs.**
Nitrate	50	P,G	Cool, 4°C	48 hrs.**

Measurement	Vol. Req.(ml.)	Container[2]	Preservative[3,4]	Holding Time[5]
Dissolved Oxygen				
Probe	300	G	None Req.	Analyze Immediately
Winkler	300	G	Fix on site and store in dark	8 hrs.
Phosphorus				
Orthophosphate, dissolved	50	G	Filter on site Cool, 4°C	48 hrs.
Total	50	G	Cool, 4°C HNO_3 to pH<2	6 mos.
Total, dissolved	50	G	Filter on site Cool, 4°C HNO_3 to pH<2	6 mos.
Silica	50	P	Cool. 4°C HNO_3 to pH < 2	6 mos.
Sulfate	50	P	Cool, 4°C	28 days
Sulfide	500	P,G	Cool, 4°C add 2 ml. zinc acetate plus NaOH to pH>9	7 days

300 SVOA Methods

GC/MS SVOA organics: BNA, PCBs, PAHs, and/or Pesticides	1,000	G only w/TefLn	Cool, 4°C	Extract within 7 days, analyze within 40 days
Pesticides/herbicides	1,000	G only w/TefLn	Cool, 4°C	Extract within 7 days, analyze within 40 days

Measurement	Vol. Req.(ml.)	Container[2]	Preservative[3,4]	Holding Time[5]
HPLC SVOA organics Ex: carbonates	1,000	G only w/TefLn	Cool, 4°C	Extract within 7 days analyze within 40 days

400 Organics

BOD	1000	P,G	Cool, 4°C	48 hrs.
COD	50	P,G	Cool, 4°C H_2SO_4 to pH<2	28 days
Oil and grease	1000	G only	Cool, 4°C HCL to pH<2	28 days
Organic carbon	25	G	Cool, 4°C H_2SO_4 or HCL to pH<2	28 days
Phenolics	500	G only	Cool, 4°C H_2SO_4 to pH<2	28 days

400 VOA Methods

Volatile organics (VOA)	2-40 ml vial w/septum caps	G only	HCL to pH < 2 Cool, 4°C	14 days
Purgeable Halocarbons only	4-40 ml vial w/septum caps		Cool, 4°C	14 days
Purgeable Aromatic Hydrocarbons	4-40 ml vial w/septum caps		Cool, 4°C HCE	14 days
Acrolein and acrylonitrite	4-40 ml vial w/septum caps		Cool, 4°C Adjust pH to 4-5	14 days

1 More specific instructions for preservation and sampling are found with each procedure as detailed in this manual. A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 31, p. 72-81 (1976) Method D-3370 and 40 CFR Part 136.

2 Plastic(P) or Glass(G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.

3 Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

4 When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such materials for transportation is responsible for ensuring such compliance. For the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

5 Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.

Holding time starts at the time the sample is collected in the field.

6 Should only be used in the presence of residual chlorine.

7 Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before the pH adjustment in order to determine whether sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and the NaOH is added to pH 12.

8 *Note: Acid preservation has been shown to rapidly degrade nitrite to nitrate. It is listed here to accomodate sites specifically requiring acid preservation in their permits. It is strongly recommended that nitrate-nitrite samples not be preserved. Holding times have been demonstrated to exceed 28 days in non-preserved groundwater samples (references available).

** Nitrate or nitrite samples only will not be collected for this project.

SAMPLE PRESERVATION (ACID/BASE) PROCEDURES

Proper Presentation (acid/base)

In order to ensure that a sufficient amount of preservative (acid or base) has been added to a sample, a method has been established for checking the pH of a sample with as little disturbance as possible to the sample. Field preservation kits (see pg. A20 and A21) are available upon request from the EML Client Services Department. These can be used, if necessary, to complete the preservation of samples.

Required equipment includes: Capillary tubes
pH paper (narrow range 0-6)
Extra preservative vials (supplied by EML)

When samples requiring preservation to a specific pH range are required, a duplicate sample will be collected for the purpose of verifying the proper preservation requirements has been attained. The duplicate sample will be subjected to the following procedure to verify pH, thereby sparing the actual environmental sample to be sent for laboratory analysis from unnecessary disturbance and possible loss of chemical species.

Once a sample has been preserved with preservative supplied by the EML, and inverted several times to mix the sample, the following procedures should be followed:

1. Open duplicate sample bottle.

2. Insert one capillary tube into the sample bottle, limiting the length of time of insertion into the sample until the tube is filled. Do not plug the end of the capillary tube. The water will rise into this tube on its own.

3. Remove the capillary tube and close the sample bottle.

4. Place the capillary tube end onto a piece of pH paper and determine the pH value.

5.
   a. If the pH is less than 2 (for acid preservation) or greater than 12 (for base preservation), the sample has been adequately preserved.

   b. If the proper pH has not been achieved, an additional preservative vial, the same type as originally used, should be emptied into the sample and mixed, and steps 1-5 should be repeated.

FIELD PRESERVATION KIT

QUANTITY	ITEM DESCRIPTION	VENDOR	CATALOG #
2 packs	Colorfast pH indicator strips Intermidiate Range (0-6 pH)	VWR	#EN-9590-3
1 pack	Kimble non-heparinzed capillary tubes (200 ct.)	VWR	#15401-537
1 pack	Pyrex glass beaker #T-1000 (150 ml.)	VWR	#13912-502
1 pack	Flambeau high density 14.5" plastic tool box	Ames	#14800
14 packs	5 ml. glass vials containing 1:8 dilution HCL	EML	
7 packs	5 ml. plastic vials containing 1:1 dilution Nitric acid	EML	
6 packs	5 ml. glass vials containing 1:1 dilution Sulphuric acid	EML	

FIELD PRESERVATION KIT ACIDIFICATION INFORMATION SHEET

METHODS	PRESERVATIVE	VIAL	VOLUME	PRESERVED pH
CMR. (all)	Sulphuric Acid	8 ml. glass	1 mls.	<2
INORG. (all)	Nitric Acid HNO_3	8 ml. plastic	2 mls.	<2
VOA	Hydrochloric Acid HCL (1:1)		.3 mls.	<2
VOA*	Hydrochloric Acid HCL (1:8)		.3 mls.	4-5

* Methods requiring pH preservation between pH 4-5

SAMPLE NUMBERING SYSTEM AND ADDITIONAL DATA REQUIRED PRIOR TO A SAMPLING EVENT

* Facility Name and Site Code

The facility name and site code must be provided and all results are reported using the facility site code rather than the facility name, to maintain confidentiality. All monitoring data in automated storage in LIMS are filed under this number. All analyses done for WMI by the EML are stored in the EML's system and transferred to LIMS under this code. Site codes are also used on all Field Chain-of-Custody Records and Field Information Forms. If a site is new or is being expanded, a site code must be obtained from the Client Services Department of the EML.

* Monitoring Program Task(s)

For regularly scheduled events, reference is given to the tasks to be included in sampling event. For unscheduled events, a new task will be assigned. Generally, all samples requiring identical analyses will be grouped under a single task.

* Number of Samples and Matrix

The number of samples to be collected and analyzed must be specified including field and trip blanks, sample types, and matrices (e.g., water, duplicates and soil). It is important to identify samples that may have a complex matrix or potential interferences, such as high sulfide or chloride or organic concentrations. These samples require special cleanup procedures prior to analysis. The categories of matrices are listed in the Appendix.

* Sample Point Designations

Sample point designations must be standardized to avoid confusion. These designations are to be limited to six characters (alphabetic and/or numeric). The EML's Account Representatives will assist clients determine sample point designation assignments for emergency situations. The designations established in LIMS are to be used by the sampling crew, by the EML, and by the subcontract lab. The EML has been instructed not to accept samples for analysis that do not contain established sample point identifiers. If a new code must be established, or if a code must be modified, contact both the Corporate Environmental staff and the EML's Client Services Department.

The EML also uses a prefix to the sample method designation code which identifies the sample type. Sample I.D.'s with the abbreviated method designation codes are printed on the bottle labels. The method designations and associated methods are listed in the Appendix.

For samples that are field duplicates, the appropriate source code with the designation code is to be used (e.g., X-DUP). The Program Manager will retain the identity of the duplicate sample.

* Method Designations

The lab group is identified with an abbreviation of lab name and methodology. This abbreviation helps differentiate various analysis requirements for a lab group's samples or program tasks. For example, with INICPDISFE: IN represents the inorganics lab group; ICP represents ICP methodology; DIS represents dissolved and FE represents the metals for which to analyze (iron).

* Sampling Date(s)/Schedule

Specific dates for sample collection are to be scheduled as soon as possible. Expected arrival dates for sample AquaPaks should be communicated to the EML Account Representative or Sampling Coordinator so the lab manager may schedule work and reserve lab capacity for the event. Regulatory agencies should also be notified of scheduled dates as required.

* Analytes to be Tested/Reporting Requirements

The analytes to be tested are to be listed by sample designations. If not previously identified, non-routine detection limits or methodologies required by a regulatory agency must be defined as special instructions.

* Required Sampling Arrangements

The sampling team must be identified. The EML's Account Representative will maintain communications with those responsible for sampling (e.g., AquaPak check, transportation arrangements), and will work with the sampling team in coordinating sampling schedules. For special events, special expertise or equipment may be required, and as much information as needed should be provided for the subcontract sampling team.

4.2.1.3 The AquaPak and Sample Bottles

The sampler or sampling team is responsible for checking all sample bottles and AquaPaks when received and for preparing ice packs prior to the sampling event. This person must immediately notify the EML of any problems or questions. The AquaPaks may also be shipped to the subcontractor or to the sampling site, depending on the location requested when the monitoring program is created. If AquaPaks are shipped to the site, the subcontractor must communicate with those on the site to ensure the timely arrival of AquaPaks. The subcontractor must also communicate with site personnel to ensure access to the site before the sampling event so that equipment, AquaPaks, and supplies may be checked. If the AquaPaks are shipped to the subcontract lab, the recipients must arrange with site's Program Manager who will contact the EML to arrange for the delivery of the AquaPaks to the sampling team. (Note: It is important that AquaPaks be sorted in buildings that do not contain solvents, cleaners or gasoline or diesel fuels.)

Bottles from more than one lab may be used when sampling. The following are some examples:

* All sample bottles and AquaPaks from the EML.
* Sample bottles and AquaPaks from both the EML and the subcontract lab.
* Bottles supplied by a regulatory agency for split samples.

The EML provides AquaPaks, which are rotary molded, high-density, plastic insulated sample containers. Each AquaPak also contains bottles packed in molded polyethene. Ice packs are provided to thermally preserve samples for up to 40 hours. Chain-of-custody seals, preservatives and forms for documentation of the sampling event, are also provided within the AquaPak.

The EML will notify the sampling team of AquaPak delivery arrangements and of the correct contents of all AquaPaks. Before the sampling event, the team leader will receive information for the sampling program on the Statement of Work (see Figure 4.4).

Upon receipt of the AquaPak, the gray numbered seal must be examined. If it is not intact, contact the EML immediately for instructions. If the seal is unbroken, break the seal, open the AquaPak, and remove the plastic field

bag from inside. (Document the seal number on the Field Chain-of-Custody Record.)

This bag contains Field Chain-of-Custody Record and Field Information Forms, red temporary seals, return labels, a Federal Express ticket, and one gray numbered seal. The gray seal is used as the final seal when the AquaPaks are returned to the lab. The red, unnumbered seals are used to seal the AquaPak if it is left unattended.

Fill out the Field Chain-of-Custody Record (Figures 4.7 and 4.7.1) and Field Information Form (Figures 4.8 and 4.8.1) when initially opening the AquaPak (e.g. record the seal number). If the samples are returned directly to the EML, send the white (original) and yellow copies of both the Field Chain-of-Custody Record and the Field Information Form with the samples. If samples are returned directly to a subcontract lab, send the pink copy of the subcontract Chain-of-Custody Record to the EML with the AquaPak.

Note the arrangement of the bottles and ice packs inside the AquaPaks before removing them. Repack them in the same order when sampling is complete. AquaPaks are packed for economy of space and often, unless special attention is given, it is difficult to fit everything back in place.

An inventory of the bottles and their condition should be noted. The bottle list, analyses required, and preservation requirements are noted on the Field Chain-of-Custody Record in the "AquaPak Contents" section. Each sample bottle is labeled with its own bottle I.D. number, which refers to the lab group, method of filtering, and necessary preservatives. All bottles used for a particular sample point must have the same bottle set number, since samples from several sample points may be packed in the same AquaPak.

New, properly cleaned sample bottles are provided in each AquaPak. The type of bottle will vary depending on the analysis required. For samples requiring preservatives, premeasured amounts of preservatives are supplied in small vials and are placed next to the proper bottle in the bottle holder. These preservatives should be added to the sample bottles after filling. However, the preservatives for the VOA and TOX methods are placed in the bottle prior to shipment. Each bottle is labeled with a sticker to identify the preservatives that are required. Preservatives must not be put in bottles that do not require them. Verification that the appropriate preservatives have been included with the bottles and that the appropriate

bottles are available is imperative. A list of sample bottles (type and size) and preservatives required for each analysis is included in the Appendix.

Each AquaPak that includes bottles for volatile organic analyses, also includes a trip blank (40 ml. vial filled with reagent free water). When volatile organic analyses have been requested, the AquaPak will contain a trip blank regardless of whether a request has been placed for analysis of the trip blank. Prior to shipment, the EML checks each trip blank vial to ensure that it has no air bubbles. Occasionally, due to pressure and temperature changes, small bubbles will appear in the blank. Note the condition of the blanks as, well as any relevant information about the AquaPak, on the Chain-of-Custody Record.

For field blank analyses, bottles will be included within the AquaPak. The water the sampling team uses in the field should be used for field blanks.

The AquaPak also contains ice packs to maintain sample temperature at the level required by EPA methods. The sampling period must be planned to allow adequate time for freezing of the ice packs. Usually a 24-hour period is required to ensure that the ice pack will be frozen solid. After the AquaPak is checked in, the ice packs should be placed in the freezer, and the Field Chain-of-Custody Record is signed. The AquaPak is then closed and a red seal is placed through the chain of custody hole. The red temporary seals are to be used whenever the AquaPak is left unattended. The gray seal is to be used after sample collection when returning the AquaPak to the EML.

If any questions arise, or if there are any problems with the AquaPaks and their contents, call the EML before proceeding.

4.2.1.4 Equipment Storage

If storage before sampling is necessary, all AquaPaks, containers, and equipment are inspected, and must be stored in a designated, contaminant-free area. (i.e. a room free of solvents, cleaners, gasoline or where none of these products are ever used.) The area must be locked. All AquaPaks must be sealed (use the red temporary seal).

Occasionally, some bottle sets will not be used (for example, if the well was dry and no sample could be taken). These sets should be returned to the lab.

4.2.2 Completing Field Records

Improper documentation or inadequate information regarding the circumstances of collection and/or subsequent disposition of the samples (e.g., field chain-of-custody) may render resultant data useless. Proper chain-of-custody documentation is a crucial part of the quality assurance and quality control (QA/QC) program. Comprehensive, consistent, and accurate documentation of field tests, measurements, and field observations is also extremely important.

Two forms must be filled out by the sampler during a sampling event. A Field Chain-of-Custody Record and a Field Information Form are completed for each sample. The original for each form must be sent with the samples to the lab. Under no circumstances will samples be analyzed without these forms. The forms are included in the client report the EML prepares when the analyses are complete. If sample bottles and AquaPaks are provided by both the EML and a subcontract lab, and each supplies Field Information Forms, only one should be filled out for each sample point. These forms must be completed with black (ball point) ink only. Pencils and felt-tip pens should not be used.

Copies of all forms are to be maintained by the Program Manager and Regional and/or Site Engineer for easy reference. Analytical data and field forms must also be maintained in the site files.

4.2.2.1 Field Chain-of-Custody Record (Figures 4.8 and 4.8.1)

To help maintain the integrity of the samples, strict chain-of-custody procedures are necessary. These procedures ensure that tampering with the samples does not occur. From the time the sample bottles leave the laboratory until the issuance of the analytical laboratory results, the samples and/or sample containers must be in the custody of assigned WMI personnel or an assigned consultant or contractor. In order to maintain the chain-of-custody, the samples must be:

* in sight of the assigned custodian;
* locked in a tamper-proof location; or
* sealed with a red, tamper-proof seal.

A written record of sample bottle possession and any transfers of samples must be maintained, and documented on the Field Chain-of-Custody Record.

The Field Chain-of-Custody Record is signed with the date and time for the following activities:

* Each time the AquaPak's seal is broken. When the AquaPak is initially opened for inspection of its contents, the seal number must also be noted.

* Each time the AquaPak is transferred to the custody of another person.

* Immediately before sealing the AquaPak for transport to the lab. (The form must be signed and enclosed within the field bag.) For the EML AquaPaks, the seal number must also be noted. Samples from the same sample point that are placed in more than one AquaPak require a Field Chain-of-Custody Record in each AquaPak.

In addition, the sample point designation, source code, date, and time (starting) for sampling must be recorded on the form. Use of prefiltration bottles and any problems with the AquaPaks' contents must also be noted on the form. Upon receipt of the AquaPak by the lab, the seal is broken, and the condition of the samples, temperature, date, and time are recorded on the Field Chain-of-Custody Record by the log-in personnel receiving the AquaPaks.

The Field Chain-of-Custody Record indicates by bottle and analysis group whether samples are to be preserved. If actual preservation and filtration procedures vary from the instructions provided in these spaces, the chain-of-custody instructions must be modified by a member of the sampling team and initialed in the appropriate locations provided on the Field Chain-of-Custody Record or on the accompanying Field Information Form. Failure to complete the Field Chain-of-Custody Record will render the data useless. Use the Chain-of-Custody Record checklist (page A-7) to ensure all pertinent information has been included.

Figure 4.8

WMI Environmental Monitoring Laboratories, Inc.

FIELD CHAIN-OF-CUSTODY RECORD

AquaPak™ PREP
AquaPak™ # _____
Date Sealed ___ / ___ / ___ (YY / MM / DD)
Seal # _____
By: _____

TE/FACILITY # ____ SITE NAME: _____

Sample Point: ____ (Source Code)

SAMPLE DATE: - ____ (YY / MM / DD)

SAMPLE TIME: ____ : ____ (2400 HR)

MATRIX CODE: _____

Water . . . . . (W) Leachate . . . . . (C)
Soil . . . . . (S) Other . . . . . (X)

Source Codes

Well . . . . . (W) Leachate System . . (C) Pretreatment Facility . . . (P) River/Stream/Brook . . (R) Soil . . . . . (S) Generation Pt . . . . (G)
Dewatering/Pressure Relief . . . . (D) Gas Condensate . . . (M) Influent . . . . . (U) Lake or Ocean . . . . . (L) Bottom Sediment . . . (B) Other . . . . . (X)
Surface Water Impoundment . . . (I) Air . . . . . (A) Effluent . . . . . (T) Outfall . . . . . (O) Noise . . . . . (N) Specify _____

ENS #

AquaPak™ CONTENT

SAMPLE I.D.	# OF BOTTLES	BOTTLE TYPE	PRESERVATIVE TYPE	ANALYTES/LAB GROUPS	FILTER Y – N	FIELD COMMENTS	E.M.L. COMMENTS
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		

CHAIN OF CUSTODY CHRONICLE

1. AquaPak™ Opened By: (print) _____ Date: ___ / ___ / ___ Time: ___ : ___ 2400 HR.
   Signature: _____ Seal #: _____ Intact: _____

I have received these materials in good condition from the above person.

2. Name: _____ Signature: _____
   Date: ___ / ___ / ___ Time: ___ : ___ 2400 HR. Remarks: _____

I have received these materials in good condition from the above person.

3. Name: _____ Signature: _____
   Date: ___ / ___ / ___ Time: ___ : ___ 2400 HR. Remarks: _____

AquaPak™ Sealed By: (print) _____ Date: ___ / ___ / ___ Time: ___ : ___ 2400 HR.
Signature: _____ Seal #: _____ Intact: _____

LAB USE ONLY
Opened By: (Signature) _____ Date: ___ / ___ / ___ Time: ___ : ___ 2400 HR.

AquaPak™ # _____ TEMP. °C _____ SEAL # _____ INTACT. _____

ORIGINAL LAB

WMI Environmental Monitoring Laboratories, Inc.

FIELD CHAIN-OF-CUSTODY RECORD

AquaPak™ # ___
Date Sealed ___ / ___ / ___ (YY / MM / DD)
Seal # 18296
By: ___

SITE/FACILITY # [illegible] SITE NAME: [illegible]

Sample Point: ___ (Source Code)

SAMPLE DATE: - ___ (YY / MM / DD)

SAMPLE TIME: ___ : ___ (2400 HR.) MATRIX CODE: ___

Water ..... (W) Leachate ..... (C)
Soil ..... (S) Other ..... (X)

Source Codes

Well ..... (W) Leachate System ... (C) Pretreatment Facility ... (P) River/Stream/Brook ... (R) Soil ..... (S) Generation Pt ..... (G)
Dewatering/Pressure Relief ..... (D) Gas Condensate ... (M) Influent ..... (U) Lake or Ocean ..... (L) Bottom Sediment ... (B) Other ..... (X)
Surface Water Impoundment ... (I) Air ..... (A) Effluent ..... (T) Outfall ..... (O) Noise ..... (N) Specify ___

ENS # [illegible]

AquaPak™ CONTENT

SAMPLE I.D.	# OF BOTTLES	BOTTLE TYPE	PRESERVATIVE TYPE	ANALYTES/LAB GROUPS	FILTER Y — N	FIELD COMMENTS	E.M.L. COMMENTS
[illegible]	01	[illegible]	[illegible]	CF	Y N		
[illegible]	01	[illegible]	NONE/COOL 4	[illegible] CR	Y N		
[illegible]	01	F	HNO3	IN	Y N		
[illegible]	01	F	HNO3	IN	Y N		
[illegible]	2	G	NONE/COOL 4	[illegible] SV	Y N		
[illegible]	2	G	NONE/COOL 4	[illegible] SV	Y N		
[illegible]	4	G	HCL	VO	Y N		
[illegible]	01	G	HCL	VO	Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		
					Y N		

CHAIN OF CUSTODY CHRONICLE

1. AquaPak™ Opened By: (print) ___ Date: ___ / ___ / ___ Time: ___ : ___ (2400 HR.)
   Signature: ___ Seal #: ___ Intact: ___

I have received these materials in good condition from the above person.

2. Name: ___ Signature: ___
   Date: ___ / ___ / ___ Time: ___ : ___ (2400 HR.) Remarks: ___

I have received these materials in good condition from the above person.

3. Name: ___ Signature: ___
   Date: ___ / ___ / ___ Time: ___ : ___ (2400 HR.) Remarks: ___

4. AquaPak™ Sealed By: (print) ___ Date: ___ / ___ / ___ Time: ___ : ___ (2400 HR.)
   Signature: ___ Seal #: ___ Intact: ___

LAB USE ONLY
Opened By: (Signature) ___ Date: ___ / ___ / ___ Time: ___ : ___ (2400 HR.)

AquaPak™ # ___ TEMP. °C ___ SEAL # ___ INTACT. ___

ORIGINAL LAB

EML FF-1A 4/90

WMI GROUNDWATER MONITORING PROGRAM
Field Chain-of-Custody Record

Field Description

Seal #*:	Enter the actual number recorded from custody seal.
ENS #*:	Enter the grouping of similar sample points and analyses together, assigned for each sampling event.
AquaPak #*:	Enter the bar coded number on the AquaPak.
Date Sealed:	Enter the date sealed at the EML for shipment.
Site/Facility #:	Enter the site code of facility being sampled.
Site Name:	Enter the complete name of the site.
Matrix Code*:	Enter water(W), soil(S), leachate(C), other(X).
Sample Point*:	Enter the source code and sample point designation for the monitoring point.
Sample Date*:	Enter the date (yy, mm, dd) monitoring point is sampled.
Sample Time*:	Enter the time sampling begins (24-hour check).
Sample I.D. Contents:	Enter the bottle I.D. for each method being run.
# of Bottles:	Enter the actual number of bottles for this bottle I.D.
Bottle Type:	Enter the type of bottle (plastic or glass).
Preservative Type:	Enter the preservative for this bottle.
Analyte/Lab Groups:	Enter the lab group performing the analysis.
Filtered*:	Enter yes or no. Should agree with the information the statement of work/bottle information.

Field Comments*: Note whether the bottle was intact, preservatives were missing, or any other problems were noticed with the bottles. Any additional problems with the AquaPaks should be noted on this form as well (e.g., the AquaPak integrity, broken chain-of-custody seal). Also, note whether filtration was done in-line, or with pressure filtration in the field or in the lab.

EML Comments: Enter any problem with receipt of bottles (e.g., empty, broken, missing, leaking) or AquaPaks.

Chain-of-Custody Chronicle*:

1. To be filled out by the person opening and checking AquaPaks contents.

2 and 3. To be filled out by someone other than the person who opened it, who had temporary custody of the AquaPaks.

4. To be filled out by the person releasing the AquaPak to the shipper, or the person who finally seals it. Names listed for 3 and 4 must be the same.

If additional space is needed, an additional Field Chain-of-Custody Record or piece of paper may be attached to the original form. Do not write on the back of the form.

* Information filled out by sampler. The other information in this section is preprinted or manually filled out at the EML.

4.2.2.2 Field Information Forms (Figures 4.9 and 4.9.1)

The Field Information Form contains information regarding site and well conditions, sampling and purging procedures used, and field measurements. The Field Information Form must be filled out by the sampler for each sample point and enclosed in the AquaPak. If sample bottles are split between the EML and the subcontract lab, the Field Information Form must be enclosed in the AquaPak transported to the EML. A Field

Information Form does not have to be completed for the subcontract lab. Field Chain-of-Custody Records must be filled out for each sample point, including dry wells. The following information must be documented:

4.2.2.2.1 Sample Point

The source code and sample point designation are contained on the Field Chain-of-Custody Record for the sample. This information will be provided prior to sampling by the EML on the Statement of Work (Figure 4.4). All sample point designations must be approved by the Corporate Environmental staff to ensure appropriate coding.

4.2.2.2.2 Purging Information

This section should be completed if the sample points are wells. All wells must be purged prior to sampling. The date and time the well was purged, the elapsed time for purging, the volume of water in the casing (gallons), and the volume purged (gallons) must be documented.

The Field Chain-of-Custody Records and Field Information Forms must indicate whether the well was dry and if a sample was not collected. The Field Information Forms must contain the purging data.

4.2.2.2.3 Purging and Sampling Information Equipment

Document the types of equipment used for purging and sample collection, and the materials of which they are constructed. If Well Wizards or pumps are used, the pump and tubing material must be documented. If a code letter does not correspond to the actual material, then a written description must be provided.

4.2.2.2.4 Samples Composited

For samples composited in the field, the procedures and proportions of samples used are to be described in the Field Comments section of the Chain-of-Custody Record and Field Information Form. (For example, "one quart obtained hourly by grab sampling over 8 hours.")

4.2.2.2.5 Field Measurements

The Site Specific Groundwater Monitoring Plan and the statement of work indicates which measurements are to be taken for each sampling event. For groundwater sampling events, the groundwater elevation (depth to

groundwater adjusted to MSL and depth to water), temperature, pH, and specific conductance at 25°C are usually required to be determined. Additional field measurements such as dissolved oxygen, field turbidity, and redox potential (EH), may also be required if noted on the Statement of Work. Space is provided for these additional parameters. Note on the document the units and values of these measurements.

4.2.2.2.6 Field Comments

The section on field comments should include field observations such as:

* Condition of the well and dedicated equipment.

* Weather condition such as wind direction and speed, upwind activities, temperature, outlook (cloud cover), barometric pressure (where needed for regulatory requirements). Any difference in weather conditions between purging and sampling should also be noted.

* Any landfill gas odor coming out of the well head vent hole.

* Sample appearance: odor, color, turbidity.

  Odor: e.g., rotten eggs, earthy, strong, moderate, slight, metallic.

  Color: True "color" is the color after the turbidity has been removed. The color observed after sampling is an "apparent color," influenced by sediment in the sample. True color may be caused by among other things, metallic ions, humus, peat, or industrial chemicals. Hold the sample up to the light and describe the color as well as possible. Observations might include no color, brown, gray or yellow.

* Turbidity:

  None: (sample is clear.);

  Trace: (sediment only slightly clouds or colors the sample; sediment does not accumulate at the bottom of the bottle.);

  Moderate: (definite cloudiness or color; sediment accumulates at the bottom of sample bottle.);

  High: (muddy appearance.).

* Reference point for well measurements.

* Well I.D. where the field blank or duplicate sample is prepared.

* All calculations for purge volumes and temperature conversions. Note when wells are purged dry, or explain when less than three casing volumes are removed.

* Duplicate field measurement results.

* Other conditions, such as sample splits with regulatory agencies, potential safety or health hazards (e.g., fire ants, presence of landfill gas in well). Note whether sampling occurred downwind from the on-site activity or disposal.

  NOTE: When samples are split with regulatory agencies, note on the Field Information Form such things as the condition of the bottles or preservatives used by the agency.

4.2.2.2.7 Sampling Certification

On the bottom of the Field Information Form, the sample team leader must sign the form certifying that the sampling procedures were in accordance with applicable federal and state regulations, and corporate policy as outlined in the WMI Manual for Groundwater Sampling and the Site-Specific Groundwater Monitoring Plans. The person signing the sampling certification must be present throughout the entire sampling process.

4.2.3 Well Observations and Measurements

It is essential that during each sampling event various observations concerning the condition of the well be made. Also required are specific measurements such as purge volume, water level, and depth of well. These observations and measurements are all documented on the Field Information Form. Any abnormal or problem condition of the well and the surrounding area must be observed and recorded on the Field Information Form upon arrival at the well location.

WMI Environmental Monitoring Laboratories, Inc.

Bottle Set: A B 8 3 8 8

FIELD INFORMATION FORM

Sample Point: (Source Code)

PURGING INFORMATION

PURGE DATE (YY MM DD) | START PURGE (2400 Hr Clock) | ELAPSED HRS | WATER VOL IN CASING (Gallons) | ACTUAL VOLUME PURGED (Gallons)

PURGING AND SAMPLING EQUIPMENT

Purging Equipment .....Dedicated | Y | | N | (circle one) Sampling Equipment .....Dedicated | Y | | N | (circle one)

Purging Device		A-Submersible Pump	D-Gas Lift Pump	G-Bailer	X- PURGING OTHER (SPECIFY)
Sampling Device		B-Peristaltic Pump	E-Venturi Pump	H-Scoop/Shovel	X- SAMPLING OTHER (SPECIFY)
		C-Bladder Pump	F-Dipper/Bottle	I-Piston Pump	
Purging Material		A-Teflon	C-Polypropylene	E-Polyethylene	X- PURGING OTHER (SPECIFY)
Sampling Material		B-Stainless Steel	D-PVC		X- SAMPLING OTHER (SPECIFY)
Tubing-Purging		A-Teflon	D-Polypropylene	F-Silicon	X- PURGING OTHER (SPECIFY)
Tubing-Sampling		B-Tygon	E-Polyethylene	G-Combination teflon/ Polypropylene	X- SAMPLING OTHER (SPECIFY)
		C-Rope X- (SPECIFY)			
Filtering Devices 0.45 μ:		A-In-line Disposable	B-Pressure	C-Vacuum	

FIELD MEASUREMENTS

Well Elevation	(ft/msl)	Land Surface Elevation	(ft/msl)
Depth to water From top of well casing	(ft)	Depth to water From land surface	(ft)
Groundwater Elevation	(ft/msl)	Groundwater Elevation	(ft/msl)
Well Depth	(ft)	Stickup	(ft)

	ph		spec. cond.		
1st	(STD)	1st	μm/cm at 25° C	Sample Temp.	(° C)
2nd	(STD)	2nd	μm/cm at 25° C	(other parameter)	value / units
3rd	(STD)	3rd	μm/cm at 25° C	(other parameter)	value / units
4th	(STD)	4th	μm/cm at 25° C	(other parameter)	value / units

FIELD COMMENTS

Sample Appearance: _____ (if applicable) Odor: _____ Color: _____ Turbidity: _____

Weather Conditions: Wind Speed _____ Direction _____ Precipitation Y/N Outlook _____

Specific Comments: _____

I certify that sampling procedures were in accordance with applicable EPA, State and WMI protocols.

___/___/___ (Date) _____ (Signature) Employer: _____

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ORIGINAL LAB

Figure 4.9.1

WMI Environmental Monitoring Laboratories, Inc.

Bottle Set: AB8388

Sample Point: WAM1 (Source Code)

FIELD INFORMATION FORM

PURGING INFORMATION

PURGE DATE (YY MM DD)	START PURGE (2400 Hr Clock)	ELAPSED HRS	WATER VOL IN CASING (Gallons)	ACTUAL VOLUME PURGED (Gallons)
910130	1315	05	56	170

PURGING AND SAMPLING EQUIPMENT

Purging Equipment ..... Dedicated (Y) | N | (Circle one) Sampling Equipment ..... Dedicated (Y) | N | (Circle one)

Purging Device	C	A-Submersible Pump	D-Gas Lift Pump	G-Bailer	X- PURGING OTHER (SPECIFY)
Sampling Device	C	B-Peristaltic Pump	E-Venturi Pump	H-Scoop/Shovel	X- SAMPLING OTHER (SPECIFY)
		C-Bladder Pump	F-Dipper/Bottle	I-Piston Pump	
Purging Material	A/B	A-Teflon	C-Polypropylene	E-Polyethylene	X- PURGING OTHER (SPECIFY)
Sampling Material	A/B	B-Stainless Steel	D-PVC		X- SAMPLING OTHER (SPECIFY)
Tubing-Purging	G	A-Teflon	D-Polypropylene	F-Silicon	X- PURGING OTHER (SPECIFY)
Tubing-Sampling	G	B-Tygon	E-Polyethylene	G-Combination teflon/ Polypropylene	X- SAMPLING OTHER (SPECIFY)
		C-Rope X- (SPECIFY)			
Filtering Devices 0.45 μ:	A	A-In-line Disposable	B-Pressure	C-Vacuum	

FIELD MEASUREMENTS

Well Elevation	* 654.01 (ft/msl)	Land Surface Elevation	(ft/msl)
Depth to water From top of well casing	99.67 (ft)	Depth to water From land surface	(ft)
Groundwater Elevation	554.34 (ft/msl)	Groundwater Elevation	(ft/msl)
Well Depth	* 134.50 (ft)	Stickup	(ft)

	ph	spec. cond. (μm/cm at 25° C)
1st	7.41 (STD)	652
2nd	(STD)	
3rd	(STD)	
4th	(STD)	

Sample Temp. 12.8 (° C)

Well dia (other parameter)	2.0 (value)	in. (units)
Stick-up (other parameter)	1.71 (value)	ft. (units)
(other parameter)	(value)	(units)

FIELD COMMENTS

Sample Appearance: (if applicable) Odor: Slight Color: Light gray Turbidity: Slight

Weather Conditions: Wind Speed 15 mph Direction Southwest Precipitation Y/N Outlook Partly cloudy

Specific Comments: *Obtained from Well I.D. Chart (1990)
Well condition is good, access good.

134.5-99.67 = 34.83 x .163 = 5.67' x 3 = 17.03
Good recharge rate; 3 casings obtained at 13:46 hrs.
Duplicate field readings: pH-7.44 at 13.0 SC-656
Well is located in N.W. corner of site-10 yds. from haul/road

I certify that sampling procedures were in accordance with applicable EPA, State and WMI protocols.

1/31/91 (Date) Michael J. Graziole (Signature) Employer: WMI/EML

EML FF-2A 4/90

ORIGINAL LAB

4.2.3.1 Observe Maintenance Conditions at Well

The conditions of the well and its surrounding area must be observed and problems and changes recorded on the Field Information Form upon arrival at the well location. The following information should be noted:

* The presence and condition of the well's identification sign.
* Whether the well was recently painted.
* Whether the well is locked and whether the key works.
* Well integrity:
    * physical surroundings (e.g., high weeds, standing water, cleanliness, nearby activities).
    * condition of the Well Wizárd or dedicated bailer.
    * condition of the protective casing.
    * any obstructions or kinks in the well casing.
    * condition of the cement footing (e.g., cracked, raised).
    * any presence of water in the annular space.
    * any grease around top of the well on the threaded caps.
    * whether the cap fits securely so contaminants cannot penetrate.
* Evidence of contamination (e.g., animal or insect parts in the well).
* Well guard posts' condition.

7. SAMPLE CUSTODY

Enseco takes pride in its ability to continually produce data that is both scientifically sound and legally defensible. In order to provide for the latter, strict custody procedures are implemented from the time the sample containers are shipped from the lab with a Bottle Chain of Custody. Enseco provides sample labels with sufficient space for the dated, original signature of the individual handling the sample. Also included are cooler seals, return shipping labels and a sample chain of custody record. See Figure 7.7. If the client provides their own sample containers, the custody procedures are implemented upon sample receipt. The custody program at Enseco involves both chain of custody documents and a computerized sample log-in and tracking system.

All samples received by Enseco are handled by the Sample Control Group. The sample control technician (SCT) meets the commercial carrier personnel and verifies the number of coolers delivered to the carrier's delivery list. The coolers are checked for radioactivity with a meter. The temperature of the coolers is measured and recorded on the sample receipt checklist, see Figure 6-1. The coolers are unpacked and the description and accompanying identification of each sample is verified against the chain of custody record submitted with the samples. The pH of all aqueous non-volatile samples which require pH adjustment for preservation is checked and recorded on the sample checklist. The preservative type is to be indicated on the sample label. The chain of custody record is signed and dated. Any discrepancies are documented and the client is notified promptly. All errors in any of the documentation is to be crossed out by a single line, dated and initialled. All Of the documentation is completed by appropriate Enseco personnel. Any discrepancies are noted on the sample checklist (Figure 6.1).

Sample delivery acceptance by Enseco is contingent upon resolution of inconsistencies in the sample chain of custody forms, breakage, receipt of sufficient documentation and project guidance regarding work orders and change orders. Samples may be rejected for these reasons plus any sample that would pose a health hazard for personnel, e.g. samples containing tetraethyl lead or radioactive substances which arrive at the lab without prior notification, are returned to the client immediately at the client's expense.

Samples then receive an Enseco five digit project code followed by a sample number. Project numbers are assigned in sequence. In formation such as client description, client number, matrix type, collection date, and Enseco receipt date are entered into a computer data base system, (LIMS) which is used to schedule analyses based on the sample holding time. The process is summarized in Figure 7.1. See Figures 7.8, 7.9, 7.10 for examples of the LIMS screens which serve as our sample log. The samples are labeled with computer generated labels stating project and sample number. The data base also provides the status for any analyses, including preparation. Samples are photographed for a permanent visual record that is entered into the project file. An acknowledgment letter is then sent to the client describing the samples received, their Enseco identification numbers, and the name of the program administrator to contact regarding the status of work being performed; a copy of the chain of custody record is included. The samples are then stored to meet all preservation criteria; a code is entered into the computer to indicate the location of the samples for the analysts.

All organic water samples are stored in temperature monitored refrigerators. Inorganic water samples are arranged by type of preservative and stored in a monitored walk-in cooler. Solids and wastes are also stored in the walk-in cooler. Sample digestates are stored on shelving units adjacent to the analytical instrumentation areas. Sample extracts are stored in temperature monitored refrigerators, designated by parameter, adjacent to the sample preparation area. All VOC samples are stored in separate refrigerators from other samples. All standards are

stored separately from samples and sample extracts. A handwritten list is kept documenting the date of disposal for all samples.

Using the LIMS system, the managers of the various chemistry departments schedule sample processing so that the holding times and turnaround requirements of the project are met. Holding times are calculated from the date of sample collection. Turnaround times are calculated from the date of sample delivery acceptance. In addition to LIMS tracking of samples, there are various preparation benchsheets and instrument logs used for tracking. (See Figures 7.11 and 7.12 for examples.)

LIMS

Enseco laboratories rely on a customized Laboratory Information Management System (LIMS) as the primary database. Client information, sample results, and QC results are all stored in the LIMS. Reports are generated directly from the database to eliminate transcription errors. A tiered security system is in place to control the ability of lab personnel to change results, and the system is designed with an audit trail that identifies when information has been changed and who changed it. The most recent two to three months of analytical data are kept on-line. All other data are archived on magnetic tape or optical disk.

Analytical tests requested by the client are grouped by the LIMS software programs, these include: analytical test, received date, collected date, and client information. Also, the number and type of bottles received for each sample and their location is generated from the program. This information can be printed in a report format so that the laboratory

MAY 22 '92 13:31 FROM ENSECO RMAL TO 17138507532 PAGE.028/049

Figure 7-1
SAMPLE ACCEPTANCE GUIDELINES

Procedures	Responsibilities SC	Responsibilities PA	Precludes SDA Yes	Precludes SDA No	Remedies
1. Coolers are received at the lab. Cooler seals are inspected.	X			X	Annotate on chain-of-custody or checklist if seals are broken.
2. Radiation levels of coolers are checked.	X		X		PA contacts client if level is above background and return samples.
3. Temperature of coolers are checked upon receipt by determining integrity of coolant (blue ice or other) and/or air temperature inside cooler.	X		X		PA contacts client if samples are frozen or received at lab above 4°C; document resolution.
4. Check for a chain-of-custody or other adequate documentation.	X		X		PA contacts client for analytical and QC requirements if no paperwork is sent with samples.
5. Sample bottles are inspected for breakage and/or leakage.	X		X		PA contacts client for broken or leaking bottles for further directions.
6. Sample bottles are inspected for appropriate labeling.		X	X		PA contacts client to resolve any discrepancies.
7. Matrices of samples are noted.		X	X		PA contacts client for discrepancies noted in matrices e.g. waters on chain-of-custody are actually wastes.
8. The chain-of-custody form is signed and dated with time and laboratory name.	X			X	

SC = Sample Control Personnel SDA = Sample Delivery Acceptance PA = Program Administration Personnel

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Figure 7-1
SAMPLE ACCEPTANCE GUIDELINES (CONTINUED)

Procedures	Responsibilities SC	Responsibilities PA	Precludes SDA Yes	Precludes SDA No	Remedies
9. The client's chain-of-custody is checked against the number and types of bottles received.		X	X		Contact client for any discrepancies noted.
10. Water VOAs are checked to see if they are filled completely.		X	X		Contact client if VOAs contain headspace or air bubbles.
11. Water VOAs are checked to see if preserved.		X	X		Contact client if not preserved. Verify that 7-day holding time test is to be used. Verify with lab that capacity exists for 7-day test.
12. Check sample bottles that are marked as filtered for sediment.		X	X		Contact client if sediment is present.
13. Check work requests for short holding time parameters.		X	X		Contact client if holding times expired in transit.
14. Check matrix specific QC requests for samples.		X	X		Contact client if insufficient volume is received and/or if the parameters requested are not appropriate for matrix specific QC.
15. Check for multi-phasic samples.		X	X		Contact client for multi-phasic samples and direction on how to proceed with work.
16. A picture of all samples is taken after they are labeled.		X		X	

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Figure 7-1
SAMPLE ACCEPTANCE GUIDELINES (CONTINUED)

Procedures	Responsibilities SC	Responsibilities PA	Precludes SDA Yes	Precludes SDA No	Remedies
17. A picture of all sub-samples is taken.	X			X	
18. Completion of sample log-in checklist		X		X	

personnel are aware of the new work. The LIMS system tracks the sample from storage through the laboratory system until the analytical process is completed and the sample is returned to the custody of the Sample Control Group for disposal. This process is summarized in Figure 7-2. LIMS privileges are assigned in the LIMS Permaint module using the privileges recommended by job function in Figure 7-3. The level of access is assigned based on the individual's level of authority and need for access.

Privileges in excess of general guidelines recommended in Figure 7-3 must have written approval by the individual's department manager or director. Approval must be documented.

Former employees of Enseco will have Permaint records with no LIMS privileges assigned.

View accounts may be created for individuals or group of individuals from another Enseco laboratory or from Corporate offices.

Data that is electronically transferred directly to the client can be provided in two ways. The data can be downloaded to a modem or it can be transferred using a floppy disc. The data that is transferred is maintained in the laboratory data base and can be regenerated if needed.

When data is electronically transferred via diskette, a checklist is completed for each disk provided to the client. The checklist documents what projects were placed on the disk and is kept in the project folder. Currently data is transferred via modem on a limited basis. Arrangements for this service and documentation are determined on a case by case basis.

Figure 7-2

ENSECO SAMPLE PROCESSING FLOW CHART

Sample Control

* Review and sign Chain-Of-Custody
* Check and document physical condition of sample
* Verify documentation and parameter assignment
* Log into LIMS
* Send acknowledgement letter to client

Proper Storage

* Store sample according to preservation guidelines
* Transfer sample to lab with proper documentation

Laboratories

* Document analytical work
* Return unused samples to Sample Control

Sample Control

* Return sample to client or arrange for sample disposal

Figure 7-3

Privileges: V - view, A - add, E - edit, R - review/release, D - delete

Job Function	LIMS Module	Privileges
USA	All modules	VAERD
USA Assistant		
Programmer/Analyst		
IS Supervisor/Manager		
Division Director	Report	VAE
Division Scientist	Project Reporting	VAE
Chief Scientist	All other modules	V
Finance and Admin. Director		
Lab Department Directors	Sample	VAERD
Lab Department Managers	Result Entry	VAER
Lab Department Supervisors	Group Code	VAERD
Principal Scientist	Group Test	VAERD
	Sample	VAERD
	Project	VAE
	Project Reporting	VAER
	Prep Result Entry	VAER
	Valid Component	VAER
	QC Result Entry	VAER
	All other modules	V
PP&C Manager/Supervisor	Shipping/Receiving	VAER
	Project	VAER
	Group Code	VAERD
	Group Test	VAERD
	Sample	VAERD
	Project	VAER
	Project Reporting	VAER
	Export/Import, Project Data	VAER
	All other modules	V
Analysts	Result Entry	VAE
	Prep Result Entry	VAER
	All other modules	V
	All other modules	V
Prep Analysts	Result Entry	VAE
	Prep Result Entry	VAER

Figure 7-3, continued

Job Function	LIMS Module	Privileges
Data Control	Sample	V E
	Result Entry	VAER
	Group Code	VAE
	Group Test	VAE
	Sample	VAE
	Project	VAE
	Project Reporting	VAER
	Prep Result Entry	VAER
	QC Result Entry	VAER
	All other modules	V
Program Administrators	Address Book	VAERD
Sales Representatives	Client	VAERD
Sales/PA Managers	Program	VAERD
	Project	VAERD
	Group Code	VAERD
	Group Test	VAERD
	Sample	VAERD
	Report	VAERD
	Project Reporting	VAER
	All other modules	V
Sample Control Supervisor	Address Book	VAE
Sample Control Coordinator	Client	VAE
	Program	VAE
	Project	VAE
	Group Code	VAE D
	Group Test	VAE D
	Sample	VAE D
	Report	VAER
	Project Reporting	VAER
	All other modules	V
Report Production	Address Book	VAE
	Report	VAE
	Project Reporting	VAER
	Group Test	VAE
	All other modules	V
QA Director	QC	VAERD
QA Specialist	QC Results Entry	VAERD
QA Chemist	Valid Component	VAER
	Project	VAE
	Report	VAER
	Project Reporting	VAER
	All other modules	V

Figure 7-3, continued

Job Function	LIMS Module	Privileges
Divisional Controller	Invoice	VAER
Accountant	Project Reporting	VAER
Accounting Clerk	All other modules	V

All new software development and software modifications shall be developed to follow a functional proposal or specification as described by the user community and reviewed by the software development team. All error reports, proposals, and specifications must be submitted to the development team by typed memorandum or VAX E-Mail.

Data entry and validation are discussed in Section 12.

All software modifications, enhancements and programs are tested by the software development team for conformance to specifications or for the elimination of the reported error.

Error corrections impacting operations or quality will be implemented immediately. If a Training database or testing system is available, it will be installed on the Training database or testing system and reviewed by the USA (for LIMS) or the submitter. If acceptable, the correction is then installed on the user (Live) system. If unacceptable, the USA or submitter communicates specific problems to the development team for correction, the correction is made (as described above), and once again it is reviewed by the USA or submitter.

Error corrections and enhancements that do not affect quality or do not significantly impact operations are sent out in periodic updates to Training databases or testing systems, if available. The corrections or enhancements are reviewed by the USA and/or users and errors or enhancements are communicated to the software development team. Modifications resulting from the review process will be processed as error corrections or short development cycle enhancements. Once the update is accepted, the user (Live) system is updated with the modifications.

Major software modifications may be implemented and reviewed independently from periodic updates but follows the same process.

Description of error corrections and minor enhancements will be sent out by the software development team to the USA's or users at the time of the software update. The USA or software development team will communicate the modifications to the users via memorandum or training sessions.

Major software modification updates will be accompanied by a training manual written by the software development team. The USA's or software development team will distribute the manual to the appropriate users and hold training sessions prior to implementing major software modifications on the user (Live) system.

Access to all Enseco laboratories is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

All exterior doors to the facility will remain locked at all times with the exception of the front entrance. During the hours of 7:00 a.m. to 6:00 p.m., the front entrance or main reception area is controlled by the receptionist and secured by locked entries. The alarm system is not activated during this time period. During the hours of 6:00 p.m. to 7:00 a.m., the front entrance is secured. The alarm system is activated during this time period to prevent all other exterior doors from being used, including sample receiving and the patio doors.

All employees and visitors are required to wear security badges at all times while on the premises of all Enseco divisions. The personnel administrator is responsible for issuing a picture I.D. badge to an employee on the first day of employment. Each employee is responsible for his/her badge. Additionally, each employee will be required to sign a "Confidentiality Agreement" which is included in the employee's personnel file. The receptionist is responsible for issuing a badge to each visitor to the facility. Visitors must request a badge from the front office of the division they visit, sign the visitor log and must be accompanied by an Enseco employee before access to any building will be allowed.

While it is not anticipated that employees will have to set or disarm the alarm system, it is important that employees understand the procedure. Unless used correctly, the alarm will go off and the Police Department will be called. This procedure is confidential and can be obtained from the Personnel Department.

For maximum security of samples when necessary (beyond the storage in the secured facility), an internal Chain-Of-Custody record is provided. analysts must sign for the samples using a special in house document (see Figure 7-4). The samples are stored in locked refrigerators.

Figure 7-4

ROCKY MOUNTAIN ANALYTICAL LABORATORY
A DIVISION OF ENSECO INC.

SAMPLE ACCOUNTABILITY RECORD
INTERNAL CHAIN OF CUSTODY DOCUMENTATION

PROJECT #_____

Samples _____ thru _____ Date Rec'd _____ Rec'd By _____ Refrigerator #_____

Sample #s	Bottle Type/Matrix	Date/Time Out	Checked Out By	Purpose	Custodian Approval	Date/Time In	Custodian Approval

An example of the Enseco Chain-Of-Custody Record used to transmit samples from the client to the laboratory is given in Figure 7-5. The Chain-Of-Custody Record (Interlaboratory Analysis Form) used to transmit samples between laboratories within Enseco is given in Figure 7-6. A copy of the Enseco Chain-of-Custody is also sent along with the Interlaboratory Chain-of-Custody. This provides the field ID#, collection date and time. If sample extracts are sent to another lab, a copy of the extraction worksheet is sent along with the sample extract.

Sample disposition is discussed in Section 6.

Laboratory Bench Sheets

Laboratory bench sheets are used to document information from routine laboratory operations, including sample preparation and analysis. Bench sheets are used to ensure that the information is recorded in a complete and organized manner and that the analysis can be reconstructed, if necessary. Portions of information from the bench sheet are also stored in the LIMS. Each bench sheet is initialed and dated as information is entered.

Laboratory Notebooks

Laboratory notebooks are used to document information that cannot easily be recorded on benchsheets such as methods development information. Each page in a laboratory notebook is initialed and dated as information is entered.

Control Charts

Enseco laboratories use control charts to visually track the LCS precision and accuracy data. These control charts are used to identify trends in the analyses which may indicate a problem with the analytical procedure. When an adverse trend is detected corrective action is performed.

Anomaly Forms

Any situation which is outside of the normal scope of operations, as described in the laboratory SOPs, is documented on an Anomaly Form. Examples of anomalous situations include: formation of a precipitate in an extract; formation of an emulsion during an extraction step; or missed holding times. These situations are documented to enable a thorough review of the data to occur.

Out-of-Control situations are also documented on Anomaly Forms. An Out-of-Control situation occurs when QC data fall outside of established control limits. The documentation associated with an Out-of-Control situation is reviewed by the supervisor and the QA Department. Out-of-Control situations trigger Corrective Action. Corrective Actions taken are also documented on the Anomaly Form.

Enseco
A Corning Company

Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002
303/421-6611 FAX: 303/431-7171

CHAIN OF CUSTODY

ENSECO CLIENT

PROJECT

SAMPLING COMPANY

SAMPLING SITE

TEAM LEADER

SAMPLE SAFE™ CONDITIONS

PACKED BY	SEAL NUMBER
SEAL INTACT UPON RECEIPT BY SAMPLING COMPANY	CONDITION OF CONTENTS
SEALED FOR SHIPPING BY	INITIAL CONTENTS TEMP °C
SEAL NUMBER	SAMPLING STATUS ☐ Done ☐ Continuing Until
SEAL INTACT UPON RECEIPT BY LAB. ☐ Yes ☐ No	CONTENTS TEMPERATURE UPON RECEIPT BY LAB. °C

DATE	TIME	SAMPLE ID/DESCRIPTION	SAMPLE TYPE	# CONTAINERS	ANALYSIS PARAMETERS	REMARKS

CUSTODY TRANSFERS PRIOR TO SHIPPING

RELINQUISHED BY (SIGNED)	RECEIVED BY (SIGNED)	DATE	TIME

SHIPPING DETAILS

DELIVERED TO SHIPPER BY		
METHOD OF SHIPMENT		AIRBILL NUMBER
RECEIVED FOR LAB	SIGNED	DATE/TIME
ENSECO PROJECT NUMBER		

White - CLIENT Pink - LAB

ENS-1133

INTERLABORATORY CHAIN OF CUSTODY

Enseco
A Corning Company

SHIP TO:

ATTENTION:

SEND RESULTS TO:

ATTENTION:

EXPORT ID	ANALYTICAL REQUESTS						SAMPLE CONDITION UPON RECEIPT	COMMENTS
TEST PRICE								
SUBTOTAL								
DISCOUNT / SURCHARGE								
TOTAL								

WRITTEN RESULTS REQUIRED BY (DATE)

VERBAL/FAC RESULTS REQUIRED BY (DATE)

P.O. No.

Q.C. ☐ STANDARD ENSECO ☐ CLP PROTOCOL ☐ PROJECT SPECIFIC

SAMPLE DISPOSAL ☐ ENSECO ☐ RETURN TO CLIENT ☐ PHONE

DETECTION LIMITS ☐ COMMON PRODUCTS ☐ OTHER*

HOLDING TIMES ☐ ENSECO ☐ EPA-CLP ☐ TIER ☐ OTHER*

RAW DATA COPIES NEEDED ☐ YES ☐ NO

CUSTODY SEALS INTACT ☐ YES ☐ NO ☐ WET WEIGHT ☐ DRY WEIGHT

*SPECIAL INSTRUCTIONS

RELINQUISHED DATE / TIME

RECEIVED DATE / TIME

Section No. /
Revision No. 1.0
Date 3/92
Page 17 of 23

FIGURE 7-7

USE BALLPOINT PEN ONLY

Company Name

Sample Description

Parameter

Date and Time

Sampler's Signature

Enseco
A Corning Company

Rocky
Mountain
Analytical
4955 Yarrow Street
Arvada, CO 80002
(303) 421-6611

Preservative
(circle one)
Acid
Base
Filtered

USE BALLPOINT PEN ONLY

Rocky Mountain Analytical

Enseco

Company Name

Sample Description

Volatile Parameter

Date Time

Sampler's Signature

FROM:

SHIP TO:

Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002

Enseco

CUSTODY SEAL

DATE

SIGNATURE

Enseco
A Corning Company

Rocky
Mountain
Analytical

CUSTODY SEAL

DATE

SIGNATURE

Enseco
A Corning Company

Rocky
Mountain
Analytical

CUSTODY SEAL

DATE

SIGNATURE

Enseco
A Corning Company

Rocky
Mountain
Analytical

FIGURE 7-8

Enseco, Inc
Wed Mar 4 15:47:08 1992 TLIGON

PROJECT (Page 1 of 2)

Client: RMAL - Name:
Program: RMAL - Name:
Project: RMAL - Name:
Type: Case: SDG: SAS: Level:
PO #: Invoice #:
Description:

Contact: RMAL - ( ) -
Alternate: RMAL - ( ) -
Rpt Cpy: RMAL - ( ) -
Ross Id: RMAL - ( ) -
Mail To: RMAL - ( ) -
Project Status: Status History: Probability: 0.00

ADD PROJECT | EDIT PROJECT | PROJECT HISTORY | PAGE 2 | MORE FUNCTIONS | GROUP CODE | SAMPLE LIST | PROGRAM

FIGURE 7-9

Enseco, Inc
Wed Mar 4 15:32:55 1992 TLIGON

PROJECT (Page 2 of 2)

Client: RMAL - Name:
Program: RMAL - Name:
Project: RMAL - Name:
Team Id: 0000 - IEC's: BG Corr's: BG Before:
Results to client computer? Deliverables:
Phone results to client? Client Project ID:
Other ENSECO lab involved? Project Duplicated from:
Subcontractor involved? Sales Exec:
Calculate holding times from collection dates? Primary PA:
Delivery Penalty: Turnaround Class:
Last Followup Date: Promised Turnaround Days: 0
Actual Start Date: Next Followup Date:
Project Due Date: Expected Start Date:
Receipt Date: Time: Storage Location:
Comments:

Log Released: Invoiced:
Cooler Shipped: Reported:
Delivery Accepted: Reviewed:
Created: Modified:

ADD PROJECT	EDIT PROJECT	PROJECT HISTORY	PAGE 1	MORE FUNCTIONS	GROUP CODE	SAMPLE LIST	PROGRAM

FIGURE 7-10

Enseco, Inc
Wed Mar 4 15:35:02 1992 TLIGON

SAMPLE DETAIL

Client: RMAL - Name:
Program: RMAL - Name:
Project: RMAL - Name:
Sample #: Aliquot: Group Code:
Sample Status:
Status History:
Client Description:
Calculate holding times from collection dates?
Collection Date: Time: Interval: 0.00
Receipt Date: Time: Turnaround Class:
Sample Due Date: Matrix:
ENSECO sample #: Permit #: Well ID:
Lab Description:
Received By:
Disposal Type: Last Instruction #: 0
Color Before: Clarity Before: Artifact:
Color After : Clarity After : Texture:
Delivery Accepted: Disposed:
Logged: Completed:
Created: Modified:

ADD SAMPLE	EDIT SAMPLE	DELETE SAMPLE	DUPLICATE SAMPLE	MORE FUNCTIONS	SAMPLE INSTRUCT.	GROUP CODE	SAMPLE LIST

FIGURE 7-11

ENSECO-ROCKY MOUNTAIN ANALYTICAL LABORATORY GC/MS INSTRUMENT: A

GC CONDITIONS								DETECTOR					
COL	PHASE DB	INJ C	INIT C	INIT TIME	RAMP RATE	FINAL C	FLOW ml/min	TYPE	EM	RANGE	MS C	QUAD C	SCAN TIME (sec)
30M	5.625	250	40	1.0 MIN.	10 C/MIN	320	CONST	MS		10^-6	200	100	

COMMENTS:

PREFIX: /ohem/T.1/
BATCH: 91.6

PROJECT	SAMPLE	ANT.	DIL/VOL	DATE (1992)	INIT	FILENAME	TAPE	IS OK	SS OK	DIL OK	COMMENTS
						.d					

FIGURE 7-12

eco - Rocky Mountain Analytical Laboratory

Project No.: _____

EXTRACTABLE ORGANICS WORKSHEET

page 1 of ___

nalyte: _____ Matrix: _____	QC Lot No.:	SCS I.D. _____ / Date Made _____ / Made by _____ / Conc. _____ / Ver. No. _____ / Amount Added _____ / Date Added _____ / Added By _____
H2Cl2 Brand _____ Lot No. _____	QC RUN NO.:	ACS I.D. _____ / Date Made _____ / Made By _____ / Conc. _____ / Ver. No. _____ / Amount Added _____ / Date Added _____ / Added By _____
cetone Brand _____ Lot No. _____	TCLP LOC NO.:	
xchange Solvent _____ Brand _____ Lot No. _____	LIMS Test Code:	Standard Addition Witnessed By _____

Sample No.	Amt. Ext. ( )	Init pH	Adj. pH	Date Ext.	Ext. By	Volume of Solvent Used	Volume of Solvent Rec.	Date Conc.	Conc. By	Solvent	Final Volume (ml)	Cleanup or Split?

omments:

Reviewed by: _____ Date: _____

STANDARD
OPERATING
PROCEDURE

Subject or Title: SAMPLE RECEIPT AND CHAIN OF CUSTODY		Page 1 of 3
SOP No.: LP-RMA-0005	Revision No.: 2.0	Effective Date: 2/27/92
Supersedes: 1.0		

ENSECO PROPRIETARY INFORMATION STATEMENT

This document has been prepared by and remains the sole property of ENSECO INC. It is submitted to a client or government agency solely for its use in evaluating Enseco's qualifications in connection with the particular project, certification or approval for which it was prepared, and is to be held proprietary to Enseco.

The user agrees by its acceptance or use of this document to return it upon Enseco's request, and not to reproduce, copy, lend or otherwise disclose or dispose of the contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically furnished. The user also agrees that where consultants or others outside of the user's organization are involved in the evaluation process, access to these documents shall not be given to those parties, unless those parties also specifically agree to these conditions.

1. Purpose:

To document receipt of all samples to the laboratory. To notify lab personnel of all incoming samples. To notify lab personnel of arriving samples that contain short holding parameters. To record the transfer of samples from the client to the lab.

Prepared by: Cindy Patterson	Date: February 27, 1992
Management Approval:	Date: 2-27-92
QA Officer Approval:	Date: 02/28/92

2. Policies:

Always assign a project number to every group of samples that arrive at the lab regardless of whether work is begun on them or not.

Project numbers are assigned in numerical order, by the LIMS system.

3. Safety:

Proper personal protective equipment must be worn, including lab coats, safety glasses and gloves.

All newly received sample coolers must be opened in an exhaust ventilation hood, and inspected for leaking samples to prevent potential exposure to suspected or unknown hazardous substances that may have vaporized during cooler transport.

If, after initial inspection and during the course of unpacking the samples from the coolers, a situation arises where leaking occurs or any samples begin to offgas or are suspected of offgassing, the sample container and cooler must be returned to the sample receiving hood.

If there are any questions, consult the Enseco Health and Safety Manual or the Health and Safety officer.

4. Procedure:

a. As samples arrive they are given a unique project number for each group of samples from one client.
b. For samples arriving by a courier check that the custody seals are intact. If broken, note this on the sample checklist. (Figure 1)
c. Sign and date the Chain of Custody (Figure 2). For samples hand delivered have the client sign and relinquish the chain of custody. Always retain the top copy with the samples and only give a bottom copy to the client.
d. Open the coolers in a hood, unpack the samples and check the information written on the chain of custody against what was received. Compare the bottles with the Bottle Chain of Custody (Figure 3) and all paperwork received. Check for the correct test, sample matrix and properly preserved bottles for each test requested. Document any discrepancies. Note any discrepancies such as missing samples, broken bottles, pH or cooler temperatures greater that 4 C on the chain of custody form and/or the sample check list. Notify the program administrator of discrepancies so that the client may be contacted in a timely manner.

e. Fill out the Sample checklist while unpacking the samples. Be certain to fill out both sides of the checklist. Check all sections.

f. Label all the samples (usually by sampling sites) with a project number and unique sample number (1,2,3,etc.). Record these numbers on the chain of custody next to the client identifications. Have a peer double check that the labelling was done correctly and referenced properly on the chain of custody.

g. Check the pH of all aqueous non-volatile samples which require pH adjustment for preservation and record on the sample checklist. Use pH strips with a range from 0-14. DO NOT DIP THE STRIPS DIRECTLY INTO THE SAMPLE. Use a disposible micro pipet to extract a small amout of sample and saturate the reactive portion of the pH strip. Compare the pH to the pH indicator colors on the box. Notify the program administrator so that the client may be contacted in a timely manner of any discrepancies (Figure 4).

h. Look for any inorganic short holding parameters and sign in these samples on the inorganic short holding notification sheet (Figure 5).

i. Take a picture of the samples. Label a manila file folder with the project number. Place the picture, sample checklist, chain of custody, Level 3 checklist (Figure 6), Bottle Chain of Custody (Figure 3) and any paperwork received in the folder.

j. Bottles needed to analyze the short holding parameters are hand delivered to the laboratory. Have an analyst sign the short hold record to document receipt of samples.

k. Place the samples in boxes and store in the walk in cooler on the appropriate team shelves pending log in.

l. After log-in, deliver the file folder to the appropriate program administrator.

m. If samples or coolers are labeled with "Quarantine Sample" stickers or other USDA labels, consult the Quarantine Sample SOP for proper handling and storage procedures.

5. Responsibilities:

Sample receiving personnel are responsible for signing the chain of custody upon receipt of samples, for knowing the location of the samples except when used by an analyst, and for signing out maximum security samples. Sample receiving personnel are responsible for noting the short holding parameters only when indicated on the paperwork from the client. Program Administrators must notify sample receiving personnel if others are to be included. Sample receiving personnel are responsible for contacting the program administrator regarding any discrepancies so that the client may be notified in a timely manner.

6. Comments:

For maximum security of samples (beyond the storage in the secured facility) an internal chain of custody is provided. Analysts must sign for the samples in a book and sign them in on return. The samples are stored in one of the locked

FIGURE 1

RMAL Industrial Sample Checklist Revision 3.0 February 27, 1992

Project #:_____ Date/Time Received:_____

Company Name & Sampling Site:_____

*Cooler #(s):_____ _____ _____ _____

Temperatures:_____ _____ _____ _____

* Place copy of airbill inside all non-RMAL coolers. Describe here.

==========

UNPACKING & LABELING CHECK POINTS:	Y	N	INITIALS
1. Radiation Checked; (record reading if > 15 mr):	___	___	_____
2. Cooler seals Intact:	___	___	_____
3. Chain of Custody Present:	___	___	_____
4. Bottles broken or leaking (comment if Y): -photograph broken bottles-	___	___	_____
5. Containers labeled (comment if N):	___	___	_____
6. pH of samples taken: -any discrepancies between pH and bottle type? (list below)_____	___	___	_____
7. Chain of Custody signed with date, time & lab:	___	___	_____
8. CoC agrees with bottle count (comment if N):	___	___	_____
9. CoC agrees with labels (comment if N):	___	___	_____
10. VOA samples filled completely (comment if N):	___	___	_____
11. VOA samples preserved:	___	___	_____
12. Sediment present in "D" bottles:	___	___	_____
13. Short holding times:	___	___	_____
14. Matrix QC verified:	___	___	_____
15. Multi phase samples present (comment if Y): -photograph multiphase samples-	___	___	_____
16. Clear picture taken & labeled:	___	___	_____

Comments: include action taken to resolve discrepancies/problems. Include a hard copy of VAX mail or extra paper if more space is needed.

_____

_____

_____

Sign and Date:_____

FIGURE 2

CHAIN OF CUSTODY

Enseco
A Corning Company

Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002
303/421-6611 FAX: 303/431-7171

ENSECO CLIENT	
PROJECT	
SAMPLING COMPANY	
SAMPLING SITE	
TEAM LEADER	

SAMPLE SAFE™ CONDITIONS

PACKED BY		SEAL NUMBER
SEAL INTACT UPON RECEIPT BY SAMPLING COMPANY		CONDITION OF CONTENTS
SEALED FOR SHIPPING BY		INITIAL CONTENTS TEMP. °C
SEAL NUMBER	SAMPLING STATUS ☐ Done ☐ Continuing Until	
SEAL INTACT UPON RECEIPT BY LAB. ☐ Yes ☐ No	CONTENTS TEMPERATURE UPON RECEIPT BY LAB. °C	

DATE	TIME	SAMPLE ID/DESCRIPTION	SAMPLE TYPE	# CONTAINERS	ANALYSIS PARAMETERS	REMARKS

CUSTODY TRANSFERS PRIOR TO SHIPPING

RELINQUISHED BY (SIGNED)	RECEIVED BY (SIGNED)	DATE	TIME

SHIPPING DETAILS

DELIVERED TO SHIPPER BY		
METHOD OF SHIPMENT		AIRBILL NUMBER
RECEIVED FOR LAB	SIGNED	DATE/TIME
ENSECO PROJECT NUMBER		

ENS-1133

White – CLIENT Pink – LAB

FIGURE 3

Enseco
A Corning Company

Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002
303/421-6611 FAX: 303/431-7171

FIGURE 3

BOTTLE ORDER CHAIN OF CUSTODY

ENS—4002

RMAL CLIENT ID	CLIENT P.O. NUMBER		INVOICE NUMBER
SHIP TO	PICK-UP ON (DATE)	TIME ☐ AM ☐ PM	DELIVERED BY (DATE)
	SHIP BY		
	UPS CHARGES	FED EX CHARGES	CLIENT FED EX ACCOUNT NUMBER
	COOLERS BILLED TO (SPECIFY CLIENT ID)		

DI _____ GALLONS | CARBON FREE _____ GALLONS | MILLI-Q _____ GALLONS

NUMBER OF BOTTLES	STANDARD WATER	PARAMETERS	NUMBER OF BOTTLES	BULK WATER	PARAMETERS
	1. 32 oz. poly (WM)	Alkalinity, BOD, Chloride, Color, Res. Chlorine, pH, Chromium (VI), Conductance, Flouride, Nitrite, MBAS, Ortho-Phos., Solids, Sulfate, Sulfite, Turbidity		20. ½ gallon glass	Bulk water analysis
	2. 16 oz. glass (BR) 50% H2SO4	Ammonia, COD, Nitrate, TKN, TON, Nitrate & Nitrite, Total Phos., TOC, Phenolics		21. 1 gallon glass	
	3. 32 oz. glass (BR) 50% H2SO4	TPH, Oil & Grease		**SOLIDS**	
	4. 16 oz. poly (WM) 20% HNO3	Metals, Hardness		30. 16 oz. glass (WM)	Organics, TPH, Metals, RAD, Oil & Grease
	5. 2-32 oz. poly (BR) 20% HNO3	Gross Alpha, Gross Beta, Uranium, Radium 226, Radium 228		31. 8 oz. glass (WM)	Wet Chem not listed for 30
	6. 8 oz. poly (WM) 50% NaOH	Total and/or Free Cyanide		32. 4 oz. glass (WM)	VOA
	7. 8 oz. poly (WM) Zn Ac & NaOH	Sulfide		**TCLP**	
	8. 4.5 oz. poly sterilize	Fecal or Total Coliform (use 2 bottles if both required)		33. 32 oz. glass (WM) 4 oz. glass (WM)	All other analytes VOA
	10. 3-40 ml glass w/septa, Na2S2O3	THM		**OTHER**	
	10A. Trip Blank				
	11. 3-40 ml glass w/septa, HCL w/out HCL	VOA, Purgeable Organics			
	11A. Trip Blank				
	12. 2-32 oz. glass (BR)	Base Neutral/Acid Compounds	BLUE ICE REQUIRED	☐ YES	☐ NO
	13. 2-32 oz. glass (BR)	Pesticides, PCBs	SPECIAL REQUIREMENTS		
	14. 32 oz. glass (BR)	Herbicides			
	15. Single: 8 oz. amber glass (BR) Quad: 32 oz. amber glass (BR) 50% H2SO4	TOX—Single: —Quad:			
			SAMPLE SAFE/COOLER NUMBERS		

REQUEST BY	DATE	TIME ☐ AM ☐ PM

RELINQUISHED BY SIGNATURE	RECEIVED BY SIGNATURE	DATE	TIME

FIGURE 4

ENSECO

pH GUIDELINE FOR SAMPLE RECEIPT

BOTTLE TYPE #	EXPECTED pH
1	neutral 5-7
2	<2
3	<2
4	<2
5	<2
6	>12
7	>9
8	neutral 5-7
10	Check done at analysis
10a	Check done at analysis
11	Check done at analysis
11a	Check done at analysis
12	Neutral 5-7
13	Neutral 5-7
14	Neutral 5-7
15	<2

Short Holding Time Board

Project Number _____ Accepted by Wet Chem Dept: _____

Client: _____ Date: _____

Date Collected: _____ Time: _____

Special Instructions: _____

FIGURE 5

Raw Data: _____

Samples
Required Analysis

Holding Time Analysis						
ASAP						
Dissolved Oxygen (O_2)						
Sulfite (SO_3^{-2})						
24 Hours						
pH (S)						
pH (Dup.)						
pH (Quad)						
Conductivity (Single)						
Conductivity (Dup.)						
Conductivity (Quad)						
Alkalinity						
Chlorine Residual						
Chromium Hex. (Cr^{+6})						
Coliform, Fecal						
Coliform, Total						
Odor						
48 Hours						
BOD						
Color						
MBAS-Surfactants						
Nitrite (NO_2^-) (Spec) or Nitrate (NO_3^-) (Tec)						
Nitrate (IC)						
Ortho-phosphate (Spec)						
Ortho-phosphate (IC)						
Turbidity						
Settleable Solids						
Other						
TDS (need conductance)						
Sulfide (O, AT)						
Ion Balance						

FIGURE 6

Level 3 Checklist

Client:_____ Project #:_____

Report Writing Checklist	Organics Yes	Organics No	Inorganics Yes	Inorganics No
1. Are departmental camera ready report cover sheets available and complete? Have anomalies been documented?	___	___	___	___
2. Have holding times been met? If no, is the holding time violation notification present?	___	___	___	___
3. Are project data sheets and QC reports complete and in a finished client report format?	___	___	___	___
4. Is the industrial report writing checklist available and complete?	___	___	___	___

Initials:_____ Date:_____

iew of Checklists, Set-up, and Project	Yes	No
1. Is the chain of custody properly signed?	___	___
2. Have all requested parameters been reported for each sample?	___	___
3. Are anomaly forms, out-of-control forms, and report writing checklists available and complete?	___	___
4. Has confirmation of final disposition of samples been generated?	___	___
5. Has the client been notified about anomalies and/or missed holding times?	___	___
6. Are ALL phone conversations documented and present in project folder? (Self sticking or gummed note pads are not appropriate.)	___	___
7. Is the report format consistent with the client's request?	___	___
8. Are the project data consistent with related measurements and parameters?	___	___
9. Has the client's overall quality objectives been met?	___	___
10. Was the quoted turnaround time met?	___	___
11. If work was subcontracted, are the results consistent with the client's request?	___	___

PA Initials:_____ Date:_____

STANDARD
OPERATING
PROCEDURE

Subject or Title: SAMPLE ARCHIVAL		Page 1 of 3
SOP No.: LP-RMA-0071	Revision No.: Original	Effective Date: Sept. 5, 1989
Supersedes: None		

1. Purpose

   To organize and maintain industrial environmental samples after analysis.

2. Policies

   2.1 Samples are stored for 30 days after analysis at no charge to the client.

   2.2 Samples may be stored at a charge of $5.00 per sample/month after the initial 30 day holding period.

   2.3 Samples may be disposed at a charge of $25.00 per sample.

3. Safety

   3.1 Lab coats, safety glasses, and gloves should be worn.

   3.2 Known hazardous samples must be segregated from other samples.

4. Procedure

   4.1 Water Samples

      4.1.1 Samples are pulled from the refrigerated cooler by the Sample Control Technician after all analysis is complete.

Prepared by: Justin Chappell	Date: August 31, 1990
Management Approval:	Date:
QA Officer Approval:	Date:

4.1.2 The samples are then brought to the sample archive area.

4.1.3 The samples are placed on numbered shelves and the laboratory identification (project) number is documented.

4.1.4 The project number is written on a shelf identification form. (Form #1).

4.1.5 These project numbers are entered into a computer that can automatically locate sample numbers and locations, the dates the samples were shelved, and the disposal codes.

4.1.6 All extracts are shelved separately.

4.1.6.1 Each week the organic extractable inventory of sample bottles and extracts is checked, and sample bottles and extracts are taken from the extraction lab to the archive area. The samples are taken to the archive area regardless of their "status." The extracts are put into bin boxes and the sample bottles are shelved.

4.1.6.2 Project numbers are tracked daily by the sample technician using a daily disposal backlog which lists project statuses.

4.1.6.3 When projects are at at status of "invoiced" the appropriate extracts are pulled from the bin boxes. They are placed on archive shelves until the 30 day holding period has expired.

4.1.6.4 "Active" extracts from the lab are placed with the appropriate active extract bottles.

4.2 Solid/Waste Samples

4.2.1 Upon completion of analysis, all solid/waste samples are archived using the same procedure as above with the exception of entering the data into the computer.

4.2.2 Sample disposition forms (Sheet #2) are sent to the client by the Program Administrator along with the acknowledgment of analysis.

4.2.3 The client can then choose to have the samples disposed of, stored or returned.

4.3 Volatiles

4.3.1 VOA vials are pulled from the volatile refrigerators when they are listed at a status of "done" on the disposal log.

4.3.2 They are shelved separately and stored until the project has been invoiced.

4.3.3 Once these samples have been invoiced, they are pulled from this shelf and put in VOA boxes until the 30 day holding period has expired.

5. Responsibilities

Sample Control Technicians are in charge of maintaining and disposing all completed environmental samples.

Enseco
A Corning Company

STANDARD
OPERATING
PROCEDURE

Subject or Title:
SAMPLE LOG-IN

SOP No.: LP-RMA-0003
Revision No.: 1.0
Effective Date: 2/27/92

Supersedes:
ORIGINAL

Personal Copies			Satellite Copies		
Name	Date Recd.	Date Returned	Location	Date Recd.	Date Returned
GOV TEAM, ENG. I, 2 + 3 TEAMS	3/2/92	1 Copy EACH			
Pipeline, UST TEAM Leaders.	↓	↓			
Mike Gallik	3/9/92	1 copy			
Richard Murphy	03/10/92				
Justin Chappel	03/17/92				
S Davis	05/11/92				

STANDARD
OPERATING
PROCEDURE

Subject or Title: SAMPLE LOG-IN		Page 1 of 3
SOP No.: LP-RMA-0003	Revision No.: 1.0	Effective Date: Feb. 27, 1992
Supersedes: Original		

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1. Purpose:

   To create analyses records in the laboratory computer for notification to lab analysts.

2. Policies:

   Log-in must be completed within 24 hours of authorization. Authorization occurs after all discrepancies are resolved or in some instances approved addendas are received.

Prepared by: Cindy Patterson	Date: February 27, 1992
Management Approval:	Date: 2-27-92
QA Officer Approval:	Date: 02/28/92

3. Safety:

Always wear gloves, safety glasses and lab coats when handling samples.

4. Procedure:

As log-in proceeds fill out the LOG-IN checklist (Figure 1) and address all the issues on this form.

4.1 Once all discrepancies (as described in the sample receipt SOP LP-RMA-0005) have been resolved as best possible, go to the second page of the project screen in LIMS. Make sure that the correct information regarding turn around time, due date, date of receipt are present. Accept the samples by using the sample delivery acceptance soft key. After each soft key, a "do" or saving key must be used to save the changes. Samples cannot be logged in unless this step is performed.

4.2 Determine group codes for each sample. Samples with the same analytical requests and same matrix type should be grouped together in one group code. Check with the program administrator regarding any special instructions for the samples.

4.3 In LIMS, go to "Group Code"by using the "Group Code" soft key. Add a group code by pressing the "Add Group Code" soft key. In the group code the LIMS sample numbers, matrix type and total sample number fields should be completed.

4.4 Next go to "Test List" using the "Test List" soft key. Use the "Add test list" soft key and add the test codes that are applicable to the client request for each sample as listed on the chain of custody and/or other supporting documentation.

4.5 Create group codes and add the requested tests for all samples with similar sample matrices and analytical requests.

4.6 Add any applicable special instructions for the corresponding groupcode in which the sample(s) is assigned. To input special instructions, the following sequence of soft keys must be used. If others are used, the special instructions will not appear. "Group Code", "Test List", "More Functions", "More Functions", "Group Instruc.", "Add Instruc." If special instructions are long, additional sequences may be necessary. Make sure a note that there are additional sequences is added.

4.7 Go to the sample list by using the "Sample List" soft key. Add each sample, (use "Add Sample" soft key) filling out all pertinent information such as; sampling date and time, group code, bottle types received. Fill out all fields.

4.8 Print the sample list, and a group code report using a control"R" at the sample list screen and group code screen. The paper work should be placed in the project folder. A level 3 checklist (Figure 2) should already be in the the project folder. Abort from this data base and go into "Project reports". Go to acceptance letter and print the acceptance letter by using the appropriate soft key. By using the appropriate soft keys print the sample description and request list paper work. This paper work should also be place into the project folder.

4.9 Fill out a chain of custody (Figure 3) for subcontracted work if necessary. A purchase order (Figure 4) must also be filled out. For subcontracting to another Enseco facility fill out the Interlaboratory Analysis Request form (Figure 5).

4.10 Perform any compositing, filtering or splitting as necessary. Create any additional preserved bottles if necessary.

4.11 Put samples in the proper locations. Volatiles are placed in separate refrigerators. Waters for organic prep are placed in refrigerators near the Organic prep labs. All other samples are placed by team identification in the walk in refrigerator.

4.12 Place project folder in the appropriate location for the team.

5. Responsibilities:

Program Administrator is responsible for reviewing that the log in has been performed correctly based upon the client's requirements and that sample receiving personnel are notified of log in errors. The Program Administrator is responsible for confirming that errors have been corrected. Sample receiving personnel are responsible for transferring the information received with the samples to the laboratory LIMS system and ensuring that a hard copy of this information is placed into the project folder.

6. Definitions:

Special instructions - Typed instructions in LIMS to the teams and analysts that are necessary to complete the work and can not be indicated by using one of the test codes assigned to the samples.

FIGURE 1

RMAL Industrial Login Review Revision 3.0 February 27, 1992

Project #: _____ Dup'd from project #: _____

Set-up By: _____ Dup'd Group Codes: _____

Logged By: _____ Date: _____

SAMPLE CONTROL REVIEW

	Y	N	INITIALS
1. Chain of Custody filled out correctly:	___	___	_____
2. Short holding time worksheet correct:	___	___	_____
3. Sample bottle/type correct:	___	___	_____
4. Overflow sample storage in special instructions:	___	___	_____
5. All login paperwork included and correct: Sample list, group code report & acceptance letter	___	___	_____
6. Trip blanks, equipment blanks, and field blanks have correct aliquot designation:	___	___	_____
7. Sample disc., request list, and acceptance letter in folder:	___	___	_____

8. Comments: Include action taken to resolve discrepancies. Include hardcopy of VAX mail, or extra paper, if more space is needed. Sign and date.

_____

_____

PROJECT ADMINISTRATOR REVIEW:

	Y	N	INITIALS	DATE
Report input sheet:	___	___	_____	_____
Invoice information:	___	___	_____	_____
All discrepancies resolved:	___	___	_____	_____
Sample and test matrices correct:	___	___	_____	_____
Sub paper work correct:	___	___	_____	_____
Clear picture of sub samples in folder	___	___	_____	_____
Special Instructions in LIMS:	___	___	_____	_____
Modified component lists checked:	___	___	_____	_____
Project due, TAT, received & collected dates OK:	___	___	_____	_____
Log released:	___	___	_____	_____

_____

_____

_____

FIGURE 2

Level 3 Checklist

Client:_____ Project #:_____

Report Writing Checklist	Organics Yes	Organics No	Inorganics Yes	Inorganics No
1. Are departmental camera ready report cover sheets available and complete? Have anomalies been documented?	___	___	___	___
2. Have holding times been met? If no, is the holding time violation notification present?	___	___	___	___
3. Are project data sheets and QC reports complete and in a finished client report format?	___	___	___	___
4. Is the industrial report writing checklist available and complete?	___	___	___	___

Initials:_____ Date:_____

Review of Checklists, Set-up, and Project	Yes	No
Is the chain of custody properly signed?	___	___
2. Have all requested parameters been reported for each sample?	___	___
3. Are anomaly forms, out-of-control forms, and report writing checklists available and complete?	___	___
4. Has confirmation of final disposition of samples been generated?	___	___
5. Has the client been notified about anomalies and/or missed holding times?	___	___
6. Are ALL phone conversations documented and present in project folder? (Self sticking or gummed note pads are not appropriate.)	___	___
7. Is the report format consistent with the client's request?	___	___
8. Are the project data consistent with related measurements and parameters?	___	___
9. Has the client's overall quality objectives been met?	___	___
10. Was the quoted turnaround time met?	___	___
11. If work was subcontracted, are the results consistent with the client's request?	___	___

PA Initials:_____ Date:_____

CHAIN OF CUSTODY

Enseco
A Corning Company

Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002
303/421-6611 FAX: 303/431-7171

ENSECO CLIENT

PROJECT

SAMPLING COMPANY

SAMPLING SITE

TEAM LEADER

SAMPLE SAFE™ CONDITIONS

PACKED BY

SEAL NUMBER

SEAL INTACT UPON RECEIPT BY SAMPLING COMPANY

CONDITION OF CONTENTS

SEALED FOR SHIPPING BY

INITIAL CONTENTS TEMP. °C

SEAL NUMBER

SAMPLING STATUS ☐ Done ☐ Continuing Until

SEAL INTACT UPON RECEIPT BY LAB ☐ Yes ☐ No

CONTENTS TEMPERATURE UPON RECEIPT BY LAB °C

DATE	TIME	SAMPLE ID/DESCRIPTION	SAMPLE TYPE	# CONTAINERS	ANALYSIS PARAMETERS	REMARKS

FIGURE 3

CUSTODY TRANSFERS PRIOR TO SHIPPING

RELINQUISHED BY (SIGNED)	RECEIVED BY (SIGNED)	DATE	TIME

SHIPPING DETAILS

DELIVERED TO SHIPPER BY

METHOD OF SHIPMENT

AIRBILL NUMBER

RECEIVED FOR LAB

SIGNED

DATE/TIME

ENSECO PROJECT NUMBER

S-1733

White – CLIENT Pink – LAB

FIGURE 4

PURCHASE ORDER

NUMBER _____

Show this Purchase Order Number on all correspondence, invoices, shipping papers and packages.

Date

Rocky Mountain Analytical Laboratory
4955 Yarrow Street, Arvada, CO 80002 (303) 421-6611

A DIVISION OF
ENSECO
INCORPORATED

To _____ Contact _____

_____ Phone _____

_____ Date Required _____

Emp. No.	Dept.	Project	Quantity	Item#/Description	Price	Amount	Rec'd.	Date

Acknowledge promptly if you are unable to ship complete order by date specified.
Invoice in triplicate

Signed by _____

INTERLABORATORY CHAIN CUSTODY

Enseco
...ning Company

PAGE OF

SHIP TO:

ATTENTION:

ANALYTICAL REQUESTS

SEND RESULTS TO:

ATTENTION:

EXPORT ID							SAMPLE CONDITION UPON RECEIPT	COMMENTS
TEST PRICE								
SUBTOTAL								
DISCOUNT / SURCHARGE								
TOTAL								

*SPECIAL INSTRUCTIONS

WRITTEN RESULTS REQUIRED BY (DATE) | VERBAL/FAC RESULTS REQUIRED BY (DATE) | P.O. No.

Q.C. ☐ STANDARD ENSECO ☐ CLP PROTOCOL ☐ PROJECT SPECIFIC

SAMPLE DISPOSAL ☐ ENSECO ☐ RETURN TO CLIENT ☐ PHONE

DETECTION LIMITS ☐ COMMON PRODUCTS ☐ OTHER*

HOLDING TIMES ☐ ENSECO ☐ EPA-CLP ☐ TIER ☐ OTHER*

RAW DATA COPIES NEEDED ☐ YES ☐ NO

CUSTODY SEALS INTACT ☐ YES ☐ NO | ☐ WET WEIGHT ☐ DRY WEIGHT

RELINQUISHED DATE / TIME

RECEIVED DATE / TIME

FIGURE 5

ENS -1109

IMPORT COPY

Enseco

STANDARD
OPERATING
PROCEDURE

Subject or Title: Laboratory Notebook Documentation		Page 1 of 2
SOP No.: LP-RMA-0021	Revision No.: 1.0	Effective Date: 12/9/87
Supersedes: Original		

1. Purpose

The laboratory notebook is a bound logbook which enables the technician/analyst to record data and notes on all laboratory work. This book is used as a reference by other staff when obtaining additional information on projects.

2. Policies

2.1 Always sign and date the bottom of every page. If the page is not completely full, draw a line through the unused portion.

2.2 Always record information in ink, never pencil.

2.3 Never use "correction fluid"; if an error is made, draw a single line through the mistake and date and initial the correction.

2.4 Never remove a page from the notebook.

2.5 Always organize the notebook in a manner that is easily read and understood by others.

3. Safety Issues

None

4. Procedure

4.1 Label the top of each page from left to right with the date, project number, analyte, and analytical method performed.

Prepared by: Gary R. Torf	Date: December 8, 1987
Management Approval: Gary Torf	Date: 5/6/88
QA Officer Approval: H Riedenbarger	Date: 5/6/88

4.2 List data in chart form when possible.

4.3 When standards are used, record all information found on the standard vial.

4.4. When solvents and other reagents are used, record the brand name, grade, lot number, and activation/deactivation dates if appropriate.

4.5 Note any problems or difficulties associated with the task you are performing.

4.6 Sign and date the bottom of every page.

5. Responsibilities

5.1 The supervisor of each laboratory department will periodically review the laboratory notebooks of their staff for completeness.

Unused AquaPaks should be returned to the EML via air courier after sampling is completed. The lab must be notified regarding the return of unused AquaPaks.

4.2.1.5 Calibration and Use of Meters

The proper measurement and documentation of field analytes are integral parts of a monitoring program. Before going to the field, all equipment must be cleaned and checked for any malfunctions.

The sampling team must calibrate all meters before using them in the field. This is necessary to ensure the proper working order of the meters, and to ensure the integrity the values measured. Manufacturers' calibration procedures must be followed. Where the manufacturer has provided no calibration procedures, follow the procedures contained in Standard Methods for the Examination of Water and Wastewater, 17th Edition.

NOTE: A COPY OF THE PROCEDURES USED IN THE CALIBRATION OF THE FIELD METERS SHOULD BE ATTACHED TO YOUR COPY OF THIS MANUAL

Calibrate the pH meter using pH 7 and pH 10 buffers, and a pH 4 buffer as a check, or with pH 7 and pH 4 buffers, and a pH 10 buffer as a check. The choice depends on the expected pH values of the samples. The calibration for pH is temperature-corrected. Please note the actual pH of your buffers at the temperature used for calibration. A chart for this is usually provided on the buffer container. You may need to calibrate the PH 7 buffer to 6.95 or 7.03 or some point in between, depending on the temperature of your buffers. This second standard should be within ±0.1 pH unit of the expected value (e.g., pH 4 buffer, calibrated to within pH 3.90-4.10). The measured value for the check buffer must be within the recalibration limits listed at the bottom of the page. If not, the meter must be recalibrated.

Calibrate the specific conductance meter standard for the conductivity expected at the site at (or converted to) 25°C. At least one additional standard must also be checked, of the same conductivity as the original standard, but cooled below room temperature (refrigerated). Checking the conductivity of this cooled standard checks the automatic temperature compensation of the meter (or conversion calculation). Specific conductance must be within ±5% of the expected value (e.g., 1,413 umhos standard, calibrated to within 1,342-1,484). If the recheck of either of the instrument's calibration does not fall within these limits, the instrument

must be recalibrated. The calibration of the field instruments must be checked every four hours and at the end of the day. Additional information on calibration of redox potential (Eh), dissolved oxygen (DO) and turbidity meters may be found in the Appendix.

If the result of the calibration check is not within the limits listed in the preceding paragraph, the meter must be recalibrated and noted in the log book. Note that the range of each well is a more important guideline.

All thermometers must also be checked, before each sampling event, for accuracy against a National Bureau of Standards (NBS) calibrated thermometer. Record this information in the Field Meter Log Book.

Sampling team members are responsible for maintaining log books for all field meters. These log books contain the same information as those for permanent lab instruments (e.g., serial number, name and model of meter, year purchased, etc.). These books also contain quality control (QC) results, maintenance performed by the factory, and calibration notes for each day the equipment is used.

NOTE: Log books may be obtained from the Senior Director of Quality Assurance for CWM for use by CWM personnel. (Refer to Figures 4.7 and 4.7.1 for examples of a log book page.)

Measure and record the specific conductance of the deionized or laboratory reagent quality water being used in the field each morning. Record the daily conductivity values in the log book of the meter that is used (field or lab). If the specific conductance of the deionized or laboratory reagent quality water is greater than 50 umhos at 25°C, the Program Manager or Site Engineer should be contacted immediately. The suspect deionized or laboratory reagent quality water should <u>not</u> be used in that case. Deionized water or reagent quality should be obtained from an alternate source (e.g., subcontract lab, EML) until the problem can be corrected.

FIELD METER LOG BOOK

Figure 4.7

		Temperature		pH Results						Specific Conductivity				Deionized Water
Date	Meter #	NBS	Meter	Read 7	Calibrate 7	Read 4	Calibrate 10	Read 4	Calibrate 4	Std.	At 25°C	At X°C	°C	Specific Conduct.

FIELD METER LOG BOOK

Figure 4.7.1 SAMPLE

		Temperature		pH Results						Specific Conductivity				Deionized Water
Date	Meter #	NBS	Meter	Read 7	Calibrate 7	Read 4	Calibrate 10	Read 4	Calibrate 4	Std.	At 25°C	At X°C	°C	Specific Conduct.
3/6/85	II	24.7°	25°	–	–	–	–	–	–	–	–	–	–	–
3/6/85	I	–	–	7.06	7.00	10.03	10.00	(check) 4.01	N/R	–	–	–	–	–
3/6/85	III	–	–	–	–	–	–	–	–	707	715	700	10°	<10
3/6/85	III	–	–	–	–	–	–	–	–	1413	1405	–	–	–
3/6/85	(backup) I(A)	24.3°	25°	7.04	7.00	10.08	10.00	(check) 4.03	N/R	707	700	723	10°	<10
3/6/85	(backup) I(A)	–	–	–	–	–	–	–	–	1413	1405	–	–	–

8. CALIBRATION PROCEDURES AND FREQUENCY

NOTE: The information in this appendix is for general information purposes only. For specific data on calibration of a particular instrument, please refer to the individual SOP.

Standard/Reagent Preparation

A critical element in the generation of quality data is the purity/quality and traceability of the standard solutions and reagents used in the analytical operations. Enseco continually monitors the quality of reagents and standard solutions through a series of well-documented procedures.

Primary reference standards and standard solutions used by Enseco are obtained from the National Institue of Standards and Technology, an EPA Cooperator Supplier, or other reliable commercial sources to ensure the highest purity possible. All standards and standard solutions are catalogued to identify the supplier, lot number, purity/concentration, receipt/preparation date, preparer's name, method of preparation, expiration date, and all other pertinent information.

Standard solutions are validated prior to use. Validation procedures can range from a check for chromatographic purity to verification of the concentration of the standard using a standard prepared at a different time or obtained from a different source. Stock and working standards are checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. Care is exercised in the proper storage and handling of standard solutions, and all containers are labeled as to compound, concentration, solvent, expiration date, and preparation data (initials of preparer/date of preparation).

Reagents are examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used; for example, every lot of dichloromethane (for organic extractables) is analyzed for undesirable contaminants prior to use in the laboratory.

Instrument Calibration and Tuning

Calibration of instrumentation is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established reporting limits. Each instrument is calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method. The frequency of calibration and calibration verification and the concentration of calibration standards are determined by the manufacturer's guidelines, the analytical method, or the requirements of special contracts.

Gas Chromatography/Mass Spectrometry (GC/MS)

Each day prior to analysis of samples, the instrument is tuned with bromofluorobenzene (BFB) for volatile compounds and decafluorotriphenylphosphine (DFTPP) for semivolatile compounds or other tune criteria as specified by the method used. No samples are analyzed until the instrument has met the tuning criteria of the method.

In general, the instrument is then calibrated for all target compounds. An initial calibration curve is produced to define the working range to establish criteria for identification. This initial calibration is evaluated on a daily basis to ensure that the system is within calibration. If the daily standard does not meet the established criteria, the system is recalibrated.

Chromatography

The field of chromatography involves a variety of instrumentation and detection systems. While calibration standards and acceptance criteria vary depending on the type of system and analytical methodology required for a specific analysis, the general principles of calibration apply uniformly. Each chromatographic system is calibrated prior to performance of analyses. Initial calibration consists of determining the working range, establishing limits of detection, and establishing retention time windows. The calibration is checked on a daily basis to ensure that the system remains within specifications. In addition, continuing calibrations are performed at frequencies required by the method used. If the calibration checks do not meet established criteria, corrective action is taken which may include recalibration and reanalysis of samples. The procedures include examination of instrument performance and analysis information, consultation with the Supervisor and a decision path to determine if recalibration and reanalysis of samples back to the previous acceptable calibration check is warranted.

Metals

Metals analysis basically involves two types of analytical instrumentation: inductively coupled argon plasma emission spectroscopy (ICP), and atomic absorption spectroscopy (AA).

Each ICP is calibrated prior to any analyses being performed using criteria prescribed in the CLP protocol. The calibration is then verified using standards from an independent source. The working range of the instrument is established once every quarter using a linear range verification check standard. No values are reported above this upper concentration value without dilution.

A calibration curve is established daily by analyzing a minimum of two standards, one of which is a calibration blank. The calibration is monitored throughout the day by analyzing a Continuing Calibration Blank (CCB) and a Continuing Calibration Verification standard (CCV). If the verification standard does not meet established criteria, corrective action must be performed. The procedures include examination of instrument performance and analysis information, consultation with the Supervisor and a decision path to determine if recalibration and reanalysis of samples back to the previously acceptable calibration check is warranted.

An interelement check standard is analyzed at the beginning and end of each analytical run, to verify that interelement and background correction factors have remained constant. Results outside of the established criteria trigger reanalysis of samples.

Each AA unit is calibrated prior to any analyses being conducted. A calibration curve is prepared with a minimum of a calibration blank and three standards and then verified with a standard that has been prepared from an independent source at a concentration near the middle of the calibration range. The calibration is then verified on an ongoing basis with a calibration blank and a midpoint calibration standard. If the ongoing calibration standard does not meet established acceptance criteria, corrective action must be performed. The procedures include examination of instrument performance and analysis information, consultation with the Supervisor and a decision path to determine if recalibration and reanalysis of samples back to the previously acceptable calibration check is warranted. All samples are spiked to verify the absence of matrix effects or interferences. The method of standard additions or sample dilution is used when matrix interferences are present.

Wet Chemistry

The field of conventional, non-metals analysis (wet chemistry) involves a variety of instrumental and wet chemical techniques. While calibration and standardization procedures vary depending on the type of system and analytical methodology required for a specific analysis, the general principles of calibration apply universally. Each system is calibrated prior to analyses being conducted. Calibration consists of defining the working range by use of a series of standard solutions, establishing limits of detection, and identifying potential interferences. The calibration is checked on an ongoing basis to ensure that the system remains within specifications. If the ongoing calibration check does not meet established criteria, corrective action must be performed. The procedures include examination of instrument performance and analysis information, consultation with the Supervisor and a decision path to determine if recalibration and reanalysis of samples back to the previous acceptable calibration check is warranted. Continuing calibrations are not performed for non-instrumental methods such as Total Dissolved Solids.

9. ANALYTICAL PROCEDURES

9.1 Background

For each entry on the Appendix IX list, EPA suggested the use of one or more SW-846 methods. SW-846 is the methods manual developed by EPA's Office of Solid Waste for compliance monitoring under RCRA. A total of 40 different methods were cited in the regulation as methods which would "provide acceptable analytical results." EPA furthermore stated that these methods were not required, nor were part of the regulation, and also stated, "some of the methods described in SW-846 are less appropriate for screening and are better suited to routine monitoring where the analyst is sure which compounds are present." Thus, laboratories were left to their own as to whether or not to use.

For most Appendix IX entries, two or more methods were cited. For example, Methods 6010 (ICP), 7040 (Flame AA) and 7041 (Furnace AA) were all listed for antimony and Methods 8010 (GC-Hall), 8020 (GC-PID), 8120 (GC-ECD) and 8270 (GC-MS) were all listed for o-dichlorobenzene. Although this array of suggested methods may provide "acceptable" results, the methods obviously do not provide equivalent results in terms of reliability, sensitivity, precision or cost.

The dilemma of selecting the appropriate methods was further complicated by the lack of method performance data in SW-846. As stated in an API report prepared by Enseco, "For some analytes e.g., allyl chloride, the analyte is not listed in the suggested methods (8010 and 8240), nor is it found listed in any other published method, although it can be analyzed by these methods. For others, e.g., acetonitrile, the suggested method is 8015, but is not listed in Method 8015. Acetonitrile is listed in Method 8030 (but not suggested in Appendix IX), however, there are no performance data for acetonitrile in Method 8030 to support the method's applicability to the analyte. Finally, for some analytes, e.g., tin, the suggested method (7870) has a very high PQL. Tin can be measured with much greater sensitivity by Method 6010, but this method is not suggested in Appendix IX for this analyte. Moreover, tin is not listed in Method 6010".

Thus, the information contained in SW-846 methods is not directly transferable to establishing a protocol. Enseco's approach to selecting appropriate methods was based on a compound by compound survey in which potential methods were selected based on well-known performance characteristics. For example, because of the high degree of reliability in identification and the acceptability by the environmental community of GC/MS methods, GC/MS was selected as a potential method for many organic compounds. Other performance characteristics evaluated included sensitivity, freedom from interferences, cost and development status of the method. For example, Method 8240 (GC/MS) was selected for acetonitrile instead of EPA's recommendation of Method 8015 (GC/FID) because of the interference problems associated with flame ionization detectors.

A primary goal was to limit the total number of methods employed so that the overall analytical costs would be reduced to the extent possible. We believed, that since this regulation was designed to detect groundwater contamination, general survey methods capable of measuring many compounds were preferable to methods which may offer somewhat better performance for a limited number of compounds in pristane matrices. If compounds were detected, then alternate methods could be utilized for ongoing monitoring efforts.

9.2 Analytical Approach

As a result of this process, 15 analytical methods were selected to establish a protocol for measuring all of the Appendix IX analytes. These methods, and the number of analytes measured by each method, are listed in Table 9-1.

Once a potential method was selected, performance data was generated for each analyte measurable by the method. The approach was as follows:

- generation of basic analytical information for identification and quantitation (retention times, mass spectra, linear range, etc.)
- evaluation of matrix bias (measured as percent recovery) in an aqueous matrix, and
- determination of a detection limit for reporting purposes.

In some cases much of this information existed. For example, this type of data was available for over 100 compounds measurable in EPA's Contract Laboratory Program. Other compounds had been studied in previous work performed by Enseco.

For each analyte Enseco established an Enseco Reporting Limit. Enseco's Reporting Limit is analogous to EPA's definition of a Practical Quantitation Limit (PQL) and is the value above which reliable data can be reported (8). These method specifications in Tables 9-2 through 9-10 list, for each method, the analytes and reporting limits and any qualifiers which could affect the interpretation of the results.

As stated in the rule, "The PQL values are in many cases based only on a general estimate for the method and not on a determination for individual compounds. PQLs are not a part of the regulation." BY comparison, the reporting limits are based on MDL studies performed at our laboratories. Our reporting limit was set equal to EPA's PQL except where our data indicated the EPA's PQL was not achievable. (i.e. our Method Detection Limit (MDL) was above EPA's PQL) or where our data indicated that EPA's PQL was wrong (i.e. the PQL was higher than the limits in SW-846 or the Contract Laboratory Program).

9.3 Sample Cleanups

Sample extract cleanups are sometimes necessary to prevent elevated reporting limits due to the presence of non-target compounds. The appropriate cleanups depend on the analytical method being used, the nature of the interferences, and other factors. Enseco's SOPs spell out circumstances under which cleanups are automatically performed (e.g. colored extract, precipitate in extract, etc.). In addition, cleanups may be performed after analysis reveals the presence of interferences. In these cases, the appropriate corrective action (e.g. cleanup) is based on the professional judgement of the analyst and laboratory supervisor. All cleanup procedures are validated prior to use.

9.4 Sample Dilutions

The Reporting Limits in Table 9-2 through 9-10 are based on the preparation of a specific sample aliquot as defined in SW-846 and summarized in Table 9-11. Any adjustments to this standard approach will result in proportionately higher reporting limits. Two factors most often impact the reporting limits, the collection of limited sample volume, and sample dilutions.

Sample dilutions are required to assure that target analytes are reportable within the working range of the calibration curve, or to reduce the level of interferences form non-target components.

In general, all decisions to dilute samples are based on a screening analysis. In any event, any sample dilutions will be discussed in the narrative of the report.

BIBLIOGRAPHY

1) Memos from the EPA; 12/4/84 by Bob Kayser Re: Appendix VIII Issues and 12/27/84 by John Skinner Re: RCRA Ground Water Monitoring Requirements.

2) "Evaluation of Ground Water Monitoring Requirements for Appendix VIII Constituents", March 15, 1986 - prepared by Enseco - Rocky Mountain Analytical Laboratory for the American Petroleum Institute.

3) "Evaluation of the Feasibility of Analyzing Groundwater for the Appendix VIII Hazardous Constituents, prepared for the American Petroleum Institute, May 1983.

4) "Evaluation of the Applicability of the SW-846 Manual to Support All RCRA Subtitle C Testing", December 20, 1984 - prepared by Enseco - Rocky Mountain Analytical Laboratory for American Petroleum Institute.

5) "Evaluation of Analytical Methods for Measuring Appendix IX Constituents in Groundwater", prepared by Enseco - Rocky Mountain Analytical Laboratory for the American Petroleum Institute API Publication 4499, July 1989).

6) "Capability of EPA Methods 624 and 625 to Measure Appendix IX Compounds," prepared by Enseco - Rocky Mountain Analytical Laboratory for the American Petroleum Institute.

7) Cornell, J.L.; Lowry, J.C.; Tilbury, M.D.; "Analysis of Selected Appendix IX Compounds in Environmental Matrices by High Performance Liquid Chromatography Particle Beam Mass Spectrometry," Annual Waste Testing and Quality Assurance Symposium, July, 1991, Washington, D.C.

8) Enseco Quality Assurance Program Plan for Environmental Monitoring, Revision 3.4, 1991.

9) Taylor, John K., Quality Assurance of Chemical Measurements, Lewis Publishers, 1987.

10) Carlberg, K.A.; Hanisch, R.C.; and Parr, J.L. - Quality Assurance in the Environmental Laboratory, Fourth Annual Waste Testing and Quality Assurance Symposium, Washington, D.C., July 1988.

TABLE 9-1
SUMMARY OF METHODOLOGY FOR MEASURING
APPENDIX IX CONSTITUENTS

SW-846 Method	Description	Technique	Preparation Method Waters	Preparation Method Soils	Number of Constituents Measured
6010	ICP Metals	ICP	3005	3050	12
7060	Arsenic	GFAA	3005	3050	1
7421	Lead	GFAA	3005	3050	1
7740	Selenium	GFAA	3005	3050	1
7841	Thallium	GFAA	3005	3050	1
7470	Mercury (Aqueous)	CVAA	*	*	1
7471	Mercury (Non-aqueous)	CVAA	*	*	
8080	Organochlorine pesticides	GC/ECD	3510	3550	31
8140	Organophosphorous pesticides	GC/NPD	3510	3550	9
8150	Organochlorine herbicides	GC/ECD	*	*	3
8240	Volatiles	GC/MS	*	*	54
8270	Semivolatiles	GC/MS	3520	3550	106
8280	Dioxins and Furans	GC/MS	*	*	7
(3)	Polar Semivolatiles	LC/MS	*	*	3
9012	Cyanide	Colorimetric	(1)	(1)	1
376.2	Sulfide	Colorimetric	*	(2)	1
	Total				232

* = The sample preparation procedure for this analysis is contained in the analytical method.
(1) = CLP Statement of Work for Inorganics, SOW Revision 2/89, Method for Total Cyanide Analysis by Midi-Distillation, Sections 7.1.1 through 7.1.11.
(2) = Proposed Update I to SW-846, 3rd Edition, Method 9030, Sections 7.1.1 through 7.1.7, Acid Soluble Sulfide.
(3) = The method is appended to this QAPP.

NOTE: This is Enseco's approach to analyzing Appendix IX compounds, based on analytical options given in SW-846 and in the Appendix IX rulemaking and method development studies performed at Enseco.

TABLE 9-2. SPECIFICATION FOR METALS AND INORGANICS

Analyte	Method	Enseco Reporting Water, mg/L	Enseco Reporting Soil, mg/kg	PQL, mg/L*
Antimony	6010	0.06	6	0.3
Arsenic	7060	0.005	0.5	0.01
Barium	6010	0.01	1	0.02
Beryllium	6010	0.002	0.2	0.003
Cadmium	6010	0.005	0.5	0.04
Chromium	6010	0.01	1	0.07
Cobalt	6010	0.01	1	0.07
Copper	6010	0.02	2	0.06
Lead	7421**	0.005	5	0.01
Mercury	7470	0.0002	0.2	0.002
Nickel	6010	0.04	4	0.05
Selenium	7740	0.005	0.5	0.02
Silver	6010	0.01	1	0.07
Thallium	7841	0.005	0.5	0.01
Tin	6010	0.1	10	8
Vanadium	6010	0.01	1	0.08
Zinc	6010	0.02	2	0.02
Cyanide	9010	0.01	0.5	0.04
Sulfide	9030	0.05	0.5	10

*Practical Quantitation Limit contained in the Appendix IX rulemaking
**Method 6010 used for soils

TABLE 9-3. SPECIFICATION FOR VOLATILE ORGANICS BY METHOD 8240

CAS #	Analyte	Enseco Reporting Limit Water ug/L	Enseco Reporting Limit Soil, ug/kg	PQL* ug/L
67-64-1	Acetone	10	10	100
75-05-8	Acetonitrile	200	200	--
107-02-8	Acrolein	100	100	5
107-13-1	Acrylonitrile	100	100	5
0107-05-1	Allyl Chloride	10	10	100
71-43-2	Benzene	5	5	5
75-27-4	Bromodichloromethane	5	5	5
75-25-2	Bromoform	5	5	5
74-83-9	Bromomethane	10	10	10
78-93-3	2-Butanone	10	10	100
75-15-0	Carbon Disulfide	5	5	5
56-23-5	Carbon Tetrachloride	5	5	5
108-90-7	Chlorobenzene	5	5	5
75-00-3	Chloroethane	10	10	10
67-66-3	Chloroform	5	5	5
74-87-3	Chloromethane	10	10	5
0126-99-8	Chloropropene	5	5	10
124-48-1	Dibromochloromethane	5	5	5
96-12-8	1,2-Dibromo-3-chloropropane	10	10	5
106-93-4	1,2-Dibromoethane	10	10	5
74-95-3	Dibromomethane	5	5	5
110-57-6	trans-1,4-Dichloro-2-butene	5	5	5
75-71-8	Dichlorodifluoromethane (Freon 12)	20	20	5
75-34-3	1,1-Dichloroethane	5	5	5
107-06-2	1,2-Dichloroethane	5	5	5
75-35-4	1,1-Dichloroethene	5	5	5
540-59-0	1,2-Dichloroethene	5	5	5
78-87-5	1,2-Dichloropropane	5	5	5
10061-01-5	cis-1,3-Dichloropropene	5	5	5
10061-02-6	trans-1,3-Dichloropropene	5	5	5
123-91-1	1,4-Dioxane	500	500	-- **
100-41-4	Ethyl Benzene	5	5	5
97-63-2	Ethyl Methacrylate	20	20	5
591-78-6	2-Hexanone	10	10	50
74-88-4	Iodomethane	5	5	5
078-83-1	Isobutanol	200	200	-- **

TABLE 9-4. SPECIFICATION FOR VOLATILE ORGANICS BY METHOD 8240

CAS #	Analyte	Enseco Reporting Limit Water ug/L	Enseco Reporting Limit Soil, ug/kg	PQL* ug/L
26-98-7	Methylacrylonitrile	5	5	
75-09-2	Methylene Chloride	5	5	5
80-62-6	Methyl Methacrylate	20	20	5
108-10-1	4-Methyl-2-pentanone	10	10	50
107-12-0	Propionitrile	5	5	5
100-42-5	Styrene	5	5	5
630-20-6	1,1,1,2-Tetrachloroethane	5	5	5
79-34-5	1,1,2,2-Tetrachloroethane	5	5	5
127-18-4	Tetrachloroethene	5	5	5
108-88-3	Toluene	5	5	5
71-55-6	1,1,1-Trichloroethane	5	5	5
79-00-5	1,1,2-Trichloroethane	5	5	5
79-01-6	Trichloroethene	5	5	5
75-69-4	Trichlorofluoromethane	5	5	5
96-18-4	1,2,3-Trichloropropane	5	5	5
108-05-4	Vinyl Acetate	10	10	5
75-01-4	Vinyl Chloride	10	10	10
1330-20-7	Xylenes (total)	5	5	5

* Practical Quantitation Limit for Method 8240 contained in the Appendix IX rulemaking

** PQL of 150 ug/L in the regulation references Method 8015; Method 8015, a GC/FID method, does not list these compounds as analytes.

TABLE 9-5. SPECIFICATION FOR SEMIVOLATILE ORGANICS BY METHOD 8270

CAS #	Analyte	Enseco Reporting Limit Water ug/L	Enseco Reporting Limit Soil, ug/kg	PQL* ug/L
83-32-9	Acenaphthene	10	330	10
208-96-8	Acenaphthylene	10	330	10
98-86-1	Acetophenone	10	330	10
53-96-3	2-Acetylaminofluorene	100	3300	10
92-67-1	4-Aminobiphenyl	10	330	10
62-53-3	Aniline	10	330	10
120-12-7	Anthracene	10	330	10
140-57-8	Aramite (4)	10	330	10
56-55-3	Benzo(a)anthracene	10	330	10
50-32-8	Benzo(a)pyrene	10	330	10
205-99-2	Benzo(b)fluoranthene	10	330	10
191-24-2	Benzo(g,h,i)perylene	10	330	10
207-08-9	Benzo(k)fluoranthene	10	330	10
100-51-6	Benzyl Alcohol	10	330	20
101-55-3	4-Bromophenyl-phenylether	10	330	10
85-68-7	Butylbenzylphthalate	10	330	10
88-85-7	2-sec-Butyl-4,6-dinitrophenol	10	330	10
106-47-8	4-Chloroaniline	10	330	20
111-91-1	bis(2-Chloroethoxy)methane	10	330	10
111-44-4	bis(2-Chloroethyl)ether	10	330	10
108-60-1	bis(2-Chloroisopropyl)ether	10	330	10
59-50-7	4-Chloro-3-methylphenol	10	330	20
91-58-7	2-Chloronaphthalene	10	330	10
95-57-8	2-Chlorophenol	10	330	10
7005-72-3	4-Chlorophenyl-phenylether	10	330	10
218-01-9	Chrysene	10	330	10
53-70-3	Dibenz(a,h)anthracene	10	330	10
132-64-9	Dibenzofuran	10	330	10
84-74-2	Di-n-butylphthalate	10	330	10
95-50-1	1,2-Dichlorobenzene	10	330	10
541-73-1	1,3-Dichlorobenzene	10	330	10
106-46-7	1,4-Dichlorobenzene	10	330	10
91-94-1	3,3'-Dichlorobenzidine	20	660	20
120-83-2	2,4-Dichlorophenol	10	330	10
87-65-0	2,6-Dichlorophenol	10	330	10
84-66-2	Diethylphthalate	10	330	10
60-11-7	p-Dimethylaminoazobenzene	10	330	10
57-97-662	7,12-Dimethylbenz(a) anthracene	10	330	10

TABLE 9.5 SPECIFICATION FOR SEMIVOLATILE ORGANICS BY METHOD 8270 (CONTINUED)

CAS #	Analyte	Enseco Reporting Limit Water ug/L	Enseco Reporting Limit Soil, ug/kg	PQL* ug/L
119-90-4	3,3'-Dimethylbenzidine	10	330	10
122-09-8	a,a-Dimethylphenethylamine	10	330	10
105-67-9	2,4-Dimethylphenol	10	330	10
131-11-3	Dimethylphthalate	10	330	10
99-65-0	1,3-Dinitrobenzene	10	330	10
534-52-1	4,6-Dinitro-2-methylphenol	50	1600	50
51-28-5	2,4-Dinitrophenol	50	1600	50
121-14-2	2,4-Dinitrotoluene	10	330	10
606-20-2	2,6-Dinitrotoluene	10	330	10
117-84-0	Di-n-octylphthalate	10	330	10
122-39-4	Diphenylamine (1)	10	330	10
117-81-7	bis(2-Ethylhexyl)phthalate	10	330	10
62-50-0	Ethyl methanesulfonate	10	330	10
206-44-0	Fluoranthene	10	330	10
86-73-7	Fluorene	10	330	10
118-74-1	Hexachlorobenzene	10	330	10
87-68-3	Hexachlorobutadiene	10	330	10
77-47-4	Hexachlorocyclopentadiene	10	330	10
67-72-1	Hexachloroethane	10	330	10
1888-71-7	Hexachloropropene	10	330	10
193-39-5	Indeno(1,2,3-c,d)pyrene	10	330	10
78-59-1	Isophorone	10	330	10
120-58-1	Isosafrole	20	660	10
91-80-5	Methapyrilene	10	330	10
56-49-5	3-Methylcholanthrene	10	330	10
66-27-3	Methyl methanesulfonate	10	330	10
91-57-6	2-Methylnaphthalene	10	330	10
95-48-7	2-Methylphenol	10	330	10
106-44-5	3/4-Methylphenol (2)	10	330	10
	Disufoton	50	1600	
	Famphur	--	----	
	Methylparathion	50	1600	

TABLE 9.5 SPECIFICATION FOR SEMIVOLATILE ORGANICS BY METHOD 8270 (CONTINUED)

CAS #	Analyte	Enseco Reporting Limit, ug/L Water ug/L	Enseco Reporting Limit, ug/L Soil, ug/kg	PQL* ug/L
91-20-3	Naphthalene	10	330	10
130-15-4	1,4-Naphthoquinone	10	330	10
134-32-7	1-Naphthylamine	10	330	10
91-59-8	2-Naphthylamine	10	330	10
88-74-4	2-Nitroaniline	50	1600	50
99-09-2	3-Nitroaniline	50	1600	50
100-01-6	4-Nitroaniline	50	1600	50
98-95-3	Nitrobenzene	10	330	10
88-75-5	2-Nitrophenol	10	330	10
100-02-7	4-Nitrophenol	50	1600	50
924-92-2	N-nitroso-di-n-butylamine	10	330	10
55-18-5	N-Nitrosodiethylamine	10	330	10
62-75-9	N-Nitrosodimethylamine	10	330	10
86-30-6	N-Nitrosodiphenylamine (1)	10	330	10
621-64-7	N-Nitroso-di-n-propylamine	10	330	10
10595-95-6	N-Nitrosomethylethylamine	10	330	10
59-89-2	N-Nitrosomorpholine	10	330	10
100-75-4	N-Nitrosopiperidine	10	330	10
930-55-2	N-Nitrosopyrrolidine	10	330	10
9-55-8	5-Nitro-o-toluidine	10	330	10
608-93-5	Pentachlorobenzene	10	330	10
76-01-7	Pentachloroethane	10	330	10
82-68-8	Pentachloronitrobenzene	50	1600	10
87-86-5	Pentachlorophenol	50	1600	50
62-44-2	Phenacetin	10	330	10
85-01-8	Phenanthrene	10	330	10
108-95-2	Phenol	10	330	10
109-06-8	2-Picoline	10	330	10
23950-58-5	Pronamide	10	330	10
129-00-0	Pyrene	10	330	10
110-86-1	Pyridine	20	660	10
94-59-7	Safrole (3)	10	330	10
	4-Nitroquinoline-1-oxide	--	--	
	Parathion	50	1600	
	4-Phenylendiamine	--	--	
	Phorate	100	3300	
	Sulfotepp	50	1600	

TABLE 9.5 SPECIFICATION FOR SEMIVOLATILE ORGANICS BY METHOD 8270 (CONTINUED)

CAS #	Analyte	Enseco Reporting Limit, ug/L Water ug/L	Soil, ug/kg	PQL* ug/L
95-94-3	1,2,4,5-Tetrachlorobenzene	10	330	10
58-90-2	2,3,4,6-Tetrachlorophenol	50	1600	10
95-53-4	2-Toluidine	10	330	10
120-82-1	1,2,4-Trichlorobenzene	10	330	10
95-95-4	2,4,5-Trichlorophenol	50	1600	10
88-06-2	2,4,6-Trichlorophenol	10	330	10
99-35-4	1,3,5-Trinitrobenzene	10	330	10

Surrogates:

4165-60-0	S1	Nitrobenzene-d5
321-60-8	S2	2-Fluorobiphenyl
1718-51-0	S3	Terphenyl-d14
4165-62-2	S4	Phenol-d5
367-12-4	S5	2-Fluorophenol
118-79-6	S6	2,4,6-Tribromophenol

Notes:

1. Cannot distinguish Diphenylamine from N-Nitroso-diphenylamine due to decomposition in the injection part.

2. 3-methyl phenol and 4-methyl phenol cannot be differentiated based on their mass spectra and retention times are identical.

3. These compounds are measured as two isomers.

4. Certain organophosphorous pesticides can be measured by Method 8270. If this option is included in an Appendix IX package, the following compounds are added to the 8270 list:

Phorate	Thionazine
Disulfoton	Dimethoate
Parathion	Famphur
Sulfotepp	Methyl parathion
o,o,o-Triethylphosphorethiote	

*Practical Quantitation Limit for Method 8270 contained in the Appendix IX rulemaking

TABLE 9.6 SPECIFICATION FOR CHLORINATED PESTICIDES AND PCB'S BY METHOD 8080

CAS #	Analyte	Enseco Reporting Limit, ug/L Water ug/L	Enseco Reporting Limit, ug/L Soil, ug/kg	PQL* ug/L
309-00-2	Aldrin	0.05	1.7	0.05
12674-11-2	Aroclor 1016	1	33	50
11104-28-2	Aroclor 1221	1	33	50
11141-16-5	Aroclor 1232	1	33	50
53469-21-9	Aroclor 1242	1	33	50
12672-29-6	Aroclor 1248	1	33	50
11097-69-1	Aroclor 1254	1	33	50
11096-82-5	Aroclor 1260	1	33	50
319-84-6	alpha-BHC	0.05	1.7	0.05
319-85-7	beta-BHC	0.05	1.7	0.05
319-86-8	delta-BHC	0.05	1.7	0.1
58-89-9	gamma-BHC (Lindane)	0.05	1.7	0.05
5103-71-9	alpha-Chlordane	0.05	1.7	0.1
5103-74-2	gamma-Chlordane	0.05	1.7	0.1
510-15-6	Chlorobenzilate	0.1	3.3	10
72-54-8	4,4'-DDD	0.1	3.3	0.1
72-55-9	4,4'-DDE	0.1	3.3	0.05
50-29-3	4,4'-DDT	0.1	3.3	0.1
2303-16-4	Diallate	1	33	10
60-57-1	Dieldrin	0.1	3.3	0.05
959-98-8	Endosulfan I	0.05	1.7	0.1
33213-65-9	Endosulfan II	0.1	3.3	0.05
1031-07-8	Endosulfan Sulfate	0.1	3.3	0.5
72-20-8	Endrin	0.1	3.3	0.1
7421-93-4	Endrin Aldehyde	0.1	3.3	0.2
76-44-8	Heptachlor	0.05	1.7	0.05
1024-57-3	Heptachlor Epoxide	0.05	1.7	1
465-73-6	Isodrin	0.1	3.3	10
143-50-0	Kepone	1	33	10
72-43-5	Methoxychlor	0.5	17	2
8001-35-2	Toxaphene	5	170	2

Surrogates: Decachlorobiphenyl, Tetrachloro-m-xylene
*Practical Quantitation Limit contained in the Appendix IX rulemaking

TABLE 9.7 SPECIFICATION FOR PHENOXY ACID HERBICIDES BY METHOD 8150

CAS #	Analyte	Enseco Reporting Limit, ug/L		PQL* ug/L
		Water ug/L	Soil, ug/kg	
94-75-7	2,4-D	1.2	24	10
93-76-5	2,4,5-T	0.20	4	2
93-72-1	Silvex	0.17	3.4	2

Surrogate: DCAA

*Practical Quantitation Limit contained in the Appendix IX rulemaking

TABLE 9-8. SPECIFICATION FOR CHLORINATED DIOXINS AND FURANS BY METHOD 8280

CAS #	Analyte	Enseco Reporting** Limit, ug/L Water ug/L	Enseco Reporting** Limit, ug/L Soil, ug/kg	PQL* ug/L
1-331	Tetrachlorodibenzodioxins (total)	0.010	0.33	0.010
1746-01-6	2,3,7,8-Tetrachlorodi-benzodioxin	0.005	0.17	0.005
1-289	Pentachlorodibenzodioxins	0.010	0.33	0.010
1-200	Hexachlorodibenzodioxins	0.010	0.33	0.010
1-332	Tetrachlorodibenzofurans	0.010	0.33	0.010
1-290	Pentachlorodibenzofurans	0.010	0.33	0.010
1-201	Hexachlorodibenzofurans	0.010	0.33	0.010

* Practical Quantitation Limit contained in the Appendix IX rulemaking
** As specified in Method 8280, reporting limits for dioxins and furans are established for each sample. The reporting limits shown are typical values.

TABLE 11. SPECIFICATION FOR CHLORINATED DIOXINS AND FURANS BY METHOD 8280

CAS #	Analyte	Enseco Reporting** Limit, ug/L Water ug/L	Soil, ug/kg	PQL* ug/L
1-331	Tetrachlorodibenzodioxins (total)	0.010	0.33	0.010
1746-01-6	2,3,7,8-Tetrachlorodi-benzodioxin	0.005	0.17	0.005
1-289	Pentachlorodibenzodioxins	0.010	0.33	0.010
1-200	Hexachlorodibenzodioxins	0.010	0.33	0.010
1-332	Tetrachlorodibenzofurans	0.010	0.33	0.010
1-290	Pentachlorodibenzofurans	0.010	0.33	0.010
1-201	Hexachlorodibenzofurans	0.010	0.33	0.010

* Practical Quantitation Limit contained in the Appendix IX rulemaking
** As specified in Method 8280, reporting limits for dioxins and furans are established for each sample. The reporting limits shown are typical values.

TABLE 12. SPECIFICATION FOR POLAR SEMIVOLATILE ORGANICS - APPENDIX IX LIST

CAS #	Analyte	Enseco Reporting Limit, ug/L Water ug/L	Enseco Reporting Limit, ug/L Soil, ug/kg	PQL* ug/L
70-30-4	Hexachlorophene	--	3300	10
56-57-5	4-Nitroquinoline-n-oxide	--	60	10
106-50-3	p-Phenylenediamine	--	1600	10

Surrogates:
p-Penylene diamine-d_4

Note: Method 8270 was recommended in the regulation. These compounds cannot be measured using Method 8270.

APPENDIX II

Quality Control Components, Spike Levels and Control Limits for Appendix IX Analytes

TABLE 14. QUALITY CONTROL COMPONENTS, SPIKE LEVELS AND CONTROL LIMITS FOR METALS AND INORGANICS

Metal	Method	Spike, mg/L	Accuracy Objective, % Recovery	Precision Objective, RPD
Antimony	6010	0.5	75-125	< 20
Arsenic	7060	0.03	75-125	< 20
Barium	6010	2.0	75-125	< 20
Beryllium	6010	0.05	75-125	< 20
Cadmium	6010	0.05	75-125	< 20
Chromium	6010	0.2	75-125	< 20
Cobalt	6010	0.5	75-125	< 20
Copper	6010	0.25	75-125	< 20
Cyanide	9010	0.20	75-125	< 20
Lead	7421	0.03	75-125	< 20
Mercury	7470	0.001	75-125	< 20
Nickel	6010	0.5	75-125	< 20
Selenium	7740	0.03	75-125	< 20
Silver	6010	0.05	75-125	< 20
Sulfide	9030	0.5	80-120	< 20
Thallium	7841	0.03	75-125	< 20
Tin	6010		75-125	< 20
Vanadium	6010	0.5	75-125	< 20
Zinc	6010	0.5	75-125	< 20

RPD = Relative Percent Difference

TABLE 15. QUALITY CONTROL COMPONENTS, SPIKE LEVELS AND CONTROL LIMITS FOR ORGANOCHLORINE PESTICIDES AND PCBs

Compound	Spike, ug/L	Accuracy Objective % Recovery	Precision Objective, RPD
Spike Components			
gamma-BHC (Lindane)	0.2	56-111	< 12
Heptachlor	0.2	50-121	< 21
Aldrin	0.2	49-109	< 16
Dieldrin	0.5	47-111	< 13
Endrin	0.5	50-123	< 16
4,4'-DDT	0.5	45-117	< 14
Surrogate Spike			
Tetrachloro-m-xylene		20-150	NA
Dibutylchlorendate		41-135	NA

NOTE: The limits shown are for water samples. Comparable limits have been established for soils. These limits change periodically based on laboratory data.

TABLE 16. QUALITY CONTROL COMPONENTS, SPIKE LEVELS AND CONTROL LIMITS FOR PHENOXYACID HERBICIDES

Compound	Spike, ug/L	Accuracy Objective, % Recovery	Precision Objective, RPD
Spike Components			
2,4-D	5.0	36-126	< 33
2,4,5-TP (Silvex)	1.0	52-135	< 28
2,4,5-T		41-158	< 34
Surrogate Spike			
DCAA	5	51-138	NA

DCAA - 2,4-Dichlorophenylacetic acid

NOTE: The limits shown are for water samples. Comparable limits have been established for soils. These limits change periodically based on laboratory data.

TABLE 17. QUALITY CONTROL COMPONENTS, SPIKE LEVELS AND CONTROL LIMITS FOR VOLATILE ORGANICS

Compound	Spike, ug/L	Accuracy Objective, % Recovery	Precision Objective, RPD
Spike Components			
1,1-Dichloroethene	50	56-138	< 20
Trichloroethene	50	76-109	< 13
Benzene	50	78-119	< 12
Toluene	50	82-114	< 13
Chlorobenzene	50	84-117	< 10
Surrogate Spikes			
1,2-dichloroethane-d_4	50	82-112	NA
4-Bromoflurorbenzene (BFB)	50	83-113	NA
Toluene-d_8	50	90-112	NA

NOTE: The limits shown are for water samples. Comparable limits have been established for soils. These limits change periodically based on laboratory data.

TABLE 18. QUALITY CONTROL COMPONENTS, SPIKE LEVELS AND CONTROL LIMITS FOR SEMIVOLATILE ORGANICS

Compound	Spike ug/L	Accuracy Objective % Recovery	Precision Objective, RPD
Spike Components			
Phenol	200	42-109	< 33
2-Chlorophenol	200	50-104	< 38
1,4-Dichlorobenzene	100	31-101	< 33
N-Nitroso-di-n-propylamine	100	49-109	< 30
1,2,4-Trichlorobenzene	100	29-100	< 33
4-Chloro-3-methylphenol	200	48-112	< 37
Acenaphthene	100	48-99	< 27
4-Nitrophenol	200	29-124	< 53
2,4-Dinitrotoluene	100	52-104	< 26
Pentachlorophenol	200	25-132	< 49
Pyrene	100	51-116	< 26
Surrogate Spikes			
Phenol-d_5	200	25-112	NA
2-Fluorophenol	200	37-100	NA
2,4,6-Tribromophenol	200	40-115	NA
Nitrobenzene-d_5	100	44-103	NA
2-Fluorobiphenyl	100	41-99	NA
Terphenyl-d_{14}	100	41-126	NA

NOTE: The limits shown are for water samples. Comparable limits have been established for soils. These limits change periodically based on laboratory data.

TABLE 19. QUALITY CONTROL COMPONENTS, SPIKE LEVELS AND CONTROL LIMITS FOR POLAR SEMIVOLATILES

Compound	Spike, ug/L	Accuracy Objectives % Recovery	Precision Objectives, RPD
Spike Components			
p-Phenylene diamine	100	30-100	<40
4-Nitroquinoline-n-oxide	100	30-100	<40
Surrogate Spikes			
p-Phenylene diamine	100	30-100	NA

NOTE: The limits shown are for water samples. Comparable limits have been established for soils. These limits change periodically based on laboratory data.

TABLE 20. QUALITY CONTROL COMPONENTS, SPIKE LEVELS AND CONTROL LIMITS FOR ORGANOPHOSPHOROUS PESTICIDES

Compound	Spike, ug/L	Accuracy Objectives % Recovery	Precision Objectives, RPD
Spike Components			
Diazinon		20-160	20
Methyl parathion		20-160	20
Malathion		20-160	20
Ethyl parathion		20-160	20
Ethion		20-160	20
Phorate		20-160	20

NOTE: The limits shown are for water samples. Comparable limits have been established for soils. These limits change periodically based on laboratory data.

STANDARD
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Subject or Title: Corrective Action Report		Page 1 of 6
SOP No.: LP-RMA-0022	Revision No.: 3.0	Effective Date: 3/25/92
Supersedes: 2.0		

1.0 Purpose

1.1 To document and communicate quality control problems encountered during analysis.

1.2 To initiate an investigation of quality control problems.

1.3 To implement corrective actions designed to ensure that quality control problems will be minimized in future analyses.

1.4 To allow follow-up of quality control problems at a later date.

2.0 Policies

2.1 All errors, deficiencies, and out-of-control situations encountered with analytical standards, client samples and laboratory-generated quality control samples must be documented on the warning/out-of-control form contained in this S.O.P.

2.2 An investigation must be initiated immediately upon discovering a problem may exist.

2.3 Upon completing the investigation, corrective actions are undertaken by, and communicated to, the appropriate team members in the team(s) in which the problem occurred.

Prepared by: Cindy Patterson	Date: 3/22/92
Management Approval: Gary Tol	Date: 3/23/92
QA Officer Approval: Gary Tol	Date: 3/23/92

2.4 The warning/out-of-control form (figure 1) must be signed and dated by any team member(s) investigating the problem, and the QC officer within the team that encountered the problem.

2.5 A copy of the signed form must be placed with the raw data and project folders of the affected samples, and provided to the Quality Assurance Department for review and archiving (it is highly recommended that the affected team also maintain a copy for communication and follow-up within the team) as described in 5.2 and 5.3.

3.0 Safety Issues

None

4.0 Procedure

4.1 Identification of Out-of-Control Situation

4.1.1 Situations which alert laboratory personnel that an out-of-control situation exists and corrective action may be necessary include, but are not limited to:

- QC data are outside the warning or acceptance limits for precision and accuracy,
- Blanks, DCS or SCS control samples contain contaminants above acceptable levels,
- There are unusual changes in detection limits,
- Surrogate recoveries in samples are consistently outside of acceptance limits,
- Deficiencies are detected by the Quality Assurance department during internal or external audits or from the results of performance evaluation samples,
- Inquiries concerning data quality are received from clients or other Enseco facilities.

4.2 Documentation of the Out-of-Control Situation

4.2.1 Briefly, but informatively describe the problem encountered on the warning/out-of-control form contained in this S.O.P. For example:

"The percent recovery for the Cyanide LCS1, QC lot 880915A, was 60%. The accuracy control limits are 75% - 125%".

Rather than,

"The LCS was out".

"The precision for Phenol in the BNA LCS, QC lot 880915A, was 66.7. The precision control limit is 42.0".

Rather than,

"The Phenol precision was no good".

"Aldrin was found in the OCP SCS, QC lot 880915A".

Rather than,

"The SCS was contaminated".

4.2.2 Specify whether the problem affected only the laboratory quality control samples, the client samples, or both.

4.3 Document the investigation of the problem.

4.3.1 List each step of the analytical procedure which was investigated, and the result/conclusion. It is acceptable to document that no definitive conclusion can be drawn, however, specific follow-up investigations will then be required as a corrective action to ensure the problem does not persist.

4.3.2 List any additional samples and/or QC lots found during the investigation to be affected by the problem.

4.4 Document the corrective actions taken to resolve the problem.

4.4.1 List any immediate steps taken to resolve the problem with the affected samples, such as repreparation or reanalysis.

4.4.2 List the actions taken to ensure that the problem does not affect future analyses. Examples include (but are not limited to): Maintenance of laboratory equipment, specific changes in standard operating procedures, additional training of personnel, and the purchasing of reagents or standards from new sources/vendors.

4.5 Communicate the resolution of the problem and any necessary follow-up to appropriate staff members.

4.5.1 The warning/out-of-control form is signed and dated by the team member investigating the problem.

4.5.2 The warning/out-of-control form is signed and dated by the QC officer of the team that encountered the problem.

4.5.3 The warning/out-of-control form is signed by a client manager (optional).

4.5.4 A copy of the warning/out-of-control form is:

- Placed with the raw data <u>and</u> project folders of any affected samples,
- Provided to the client manager when requested.
- Filed for the team by the QC officer so that trends can be monitored.
- Given to the QA Team.

4.5.5 The problem and it's resolution is communicated to additional staff members in the affected team(s) at the next scheduled group meeting.

5.0 Responsibilities

5.1 The staff member (technician, analyst/chemist, team leader, quality assurance personnel, client manager, technical director, et al) who encounters an error, deficiency or out-of-control situation is responsible for immediately documenting the problem as described in 4.2 above.

- Documentation of corrective actions,
- Ensuring corrective actions are implemented and appropriate follow-up is undertaken,
- Ensuring that a copy of the warning/out-of-control form is placed with the raw data and provided to the appropriate personnel as described in 4.5.5 and 4.5.6 above and,
- Communicating the problem and its resolution to additional team members.

5.2 The Team's QC Officer where the problem occurred is responsible for:

- The timely investigation of the problem,
- The review of the documentation of corrective actions,
- Notification to the Quality Assurance Team and affected client manager(s),

5.3 The Quality Assurance Team is responsible for:

- The timely approval of any S.O.P. changes necessitated by corrective actions.
- The timely review of all out-of-control forms for completeness and accuracy.

5.4 The client contact is responsible for communicating to the client the affect a problem may have on the analysis of their samples (verbally and/or in the final report).

6.0 Comments

6.1 All teams should encourage the use of the warning/out-of-control form by:

- Reiterating that Quality Control is the responsibility of all staff members and,

- The prompt addressing of major and minor problems brought to their attention.

FIGURE 1

WARNING/OUT OF CONTROL FORM

QC Category_____ QC Lot_____

Matrix: Aqueous / Soil (circle one) QC Run_____

PROBLEM (Be specific)

_____

_____

_____

_____

_____

ANALYST_____ DATE_____

INVESTIGATION/STEPS- RESULTS

_____

_____

_____

_____

ANALYST_____ DATE_____

CORRECTIVE ACTION TAKEN

_____

_____

_____

ANALYST_____ DATE_____

REVIEW/COMMENTS

_____

_____

TEAM QC OFFICER_____ DATE_____

OTHER OBSERVATIONS

_____

_____

_____

ANALYST_____ DATE_____

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-1
SOP Number: LM-RMA-1060
Title: Total Dissolved Solids

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

Section 5.1: A 500 ml sample will be collected, and the sample will be filtered in the field.

Section 8.1.1: Add that samples will be filtered as soon as the lab receives them.

Section 8.2.1: The information contained in this section can also be considered as a part of the preparation procedure.

Section 11.2: ND = Not Detected. The reporting limit will be indicated as a part of the report.

STANDARD
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Subject or Title:		Page 1 of 11
Total Dissolved Solids (TDS)		
SOP No.: **LM-RMA-1060**	Revision: **4.0**	Effective Date: **May 21, 1991**

Supersedes: 3.0

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1. Scope and Application

1.1 Analytes

This method is used for the determination of Total Dissolved Solids, also known as Filterable Residue.

1.2 Reporting Limits

The method reporting limit is 10 mg/L.

1.3 Applicable Matrices

This method is applicable to drinking, surface, and saline waters, domestic, and industrial wastes.

Prepared by	Date:
Tom J Stovall	May 22, 1991
Management Approval:	Date:
David B Roberts	5-22-91
QA Officer Approval:	Date:
Bill Shaw	05/22/91

1.4 Dynamic Range

This method covers the range from 10 mg/L and up. It is a gravimetric method and there is no upper limit. As a practical matter, the final residue weight should be limited to about 200 mg.

1.5 Analysis Time

The analytical time is about 30 minutes per sample, not including drying time. The total drying time is variable and can be as long as 24 to 48 hours.

2. Method Summary

A well-mixed sample is filtered through a glass fiber filter. The filtrate is evaporated at 103°C and dried to a constant weight at 180°C.

3. Comments

3.1 Interferences

3.1.1 Some samples may contain hygroscopic salts which require prolonged drying, desiccation, and rapid weighing.

3.1.2 Samples containing high concentrations of bicarbonate may require careful prolonged drying to ensure that all the bicarbonate is converted to carbonate.

3.1.3 Too much residue in the pans will crust over, entrapping water that will not be driven off during drying. Total residue should be limited to about 200 mg.

3.1.4 Oil and grease in the samples will cause unreliable results due to difficulty in drying to constant weight.

3.1.5 Some samples may have fine suspended solids which pass through the glass fiber filter causing high results.

3.1.6 Aluminum pans should not be used. Components in some samples may react to form aluminum compounds causing unreliable results.

3.2 Helpful Hints

3.2.1 Conductance multiplied by a factor between 0.55 and 0.81 gives a rough approximation of TDS. The exact relationship depends on the compounds present in the samples and may not hold for very high concentrations or samples containing nonionic species or samples with conductances greater than 10,000 umho/cm or less than 50 umho/cm.

3.2.2 The filtrate from the TSS procedure (LM-RMA-1062) may be used for TDS determination.

4. Safety Issues

Normal laboratory safety procedures should be followed. There are no special concerns with this method.

5. Sample Collection, Preservation, Containers and Holding Times

5.1 Samples are to be collected in plastic bottles. A minimum of 100 mL is required and may or may not be filtered in the field.

5.2 Refrigerate all samples to 4°C.

5.3 The holding time for TDS is 7 days.

6. Apparatus

6.1 Glass fiber filter disks, 47 mm (Gelman Type A/E). The filters should be rinsed with deionized water and dried before use. Assemble the filtration apparatus, turn on the vacuum pump, and place a filter on the holder. Rinse the filter with three 20 mL portions of deionized water, dry at 103°C and store in a desiccator.

6.2 Vacuum filtration apparatus.

6.3 Vacuum pump equipped with moisture trap.

6.4 Porcelain evaporating dishes. These must be thoroughly cleaned, rinsed with deionized water, and baked at 180°C for at least one hour before use. Store in a desiccator.

6.5 Desiccators providing space for storage of samples in process separate from filters and evaporating dishes.

6.6 Drying ovens set at 103°C and 180°C. Separate ovens should be maintained at the appropriate temperatures if possible.

6.7 Steam bath

6.8 Thermometers, NBS traceable.

6.9 Analytical balance capable of measurements to 0.0001 g.

6.10 Conductivity meter and associated apparatus.

6.11 Tweezers, graduated cylinders, and other general laboratory apparatus. Glassware must be thoroughly washed with detergent and rinsed with deionized water.

7. Reagents and Standards

7.1 Deionized water (ASTM Type II or equivalent)

7.2 All reagents and standards required for measurement of conductance (see SOP LM-RMA-1062).

7.3 DCS Solution

Use an ERA Minerals Sample or a sample from another reliable source.

8. Procedure

8.1 Preparation

8.1.1 Check samples for special instructions, making copies to include with the data package. Obtain samples from sample receiving. Fill out the benchsheet. See example benchsheet.

8.1.2 Measure and record the conductance of each sample before analysis. An accurate value, corrected for temperature and cell constant, must be obtained. If the conductance has already been measured, this value may be used. Check the Short Holding Time Sheets; TDS has been placed on them in order to identify all samples requiring conductivity. **THE CONDUCTANCE MUST BE RECORDED ON THE BENCH SHEET FOR EACH SAMPLE.**

8.1.3 If samples are visibly oily, this should be noted on the benchsheet.

8.1.4 Mark the evaporating dishes with appropriate identification. Weigh dishes for each sample to the nearest 0.0001 g. Only use dishes which have previously been baked at 180°C and stored in a desiccator.

8.1.5 Determine the proper volume of each sample to be analyzed.

8.1.5.1 If the sample has a conductance less than 3,000 umho/cm, 100 mL should be used.

8.1.5.2 If the conductance is 3,000 to 10,000 umho/cm, use 20 mL of sample. Dilute volumes of <100 mL to 100 mL final volume with deionized water.

8.1.5.3 If the conductance is extremely high (more than about 10,000 umho/cm), smaller volumes may be used. Consult your supervisor.

8.1.5.4 If there is limited sample volume, a smaller amount of sample must be analyzed. This must be noted on an anomaly sheet.

8.2 Analysis

8.2.1 Thoroughly rinse the entire filtration apparatus with deionized water before filtering each sample. Assemble the filtration apparatus and turn on the vacuum pump. Only use filters which have been previously rinsed, dried, and stored in a desiccator. The patterned side of the filter must face down.

8.2.3 Shake the sample vigorously, measure the required volume into a graduated cylinder, and rapidly transfer to the filtration apparatus. All samples are to be filtered, whether or not they have been filtered in the field.

NOTE: If the sample contain large amounts of insoluble material, a portion may be poured off after mixing and allowed to settle or an aliquot maybe taken and centrifuged, before filtering. Do not pour directly

from the bottle without mixing. This will alter the sample composition and adversely affect subsequent analyses.

8.2.4 After the sample has been filtered, rinse the graduated cylinder into the filtration apparatus with three 10 mL portions of deionized water. Continue vacuum for three minutes after filtration is complete.

8.2.5 Transfer the filtered sample to the preweighed evaporating dish. Rinse the receiving flask into the dish to ensure complete transfer of the sample.

8.2.6 Place the samples in the drying oven set at 103°C and evaporate to dryness. Record the date and time that the samples were placed in the oven and the temperature of the oven on the benchsheet. Alternately, the samples may be placed on a steam bath and evaporated to dryness.

8.2.7 Prepare new filters and dishes to replace the ones you have used. Instructions are given in Sections 6.1 and 6.4 respectively.

8.2.8 When the samples are dry, transfer to the drying oven set at 180 +/- 2°C and heat for a minimum of one hour. If a number of samples are being dried simultaneously, additional time may be necessary. Record the date and time that the samples are placed in the drying oven. In addition, record the oven temperature on the bench sheet at the beginning and end of the drying period.

8.2.9 Cool the samples in a desiccator for two to three hours. Remove and weigh one tray crucibles at a time to prevent absorption of moisture. Record the date and time of this measurement on the bench sheet.

8.2.10 Return the samples to the oven and bake for another hour. Cool the samples in a desiccator and weigh. Record the date, time, and oven temperatures as above. Repeat this cycle again if the weight changes by more than 1.0 mg. If a constant weight is not achieved within three drying cycles, note this on an anomaly sheet. Do not continue the drying, cooling, weighing cycle beyond three times.

8.3 **Conclusion**

8.3.1 Clean all glassware, apparatus and the work area.

8.3.2 Return all samples to sample receiving.

8.3.3 Calculate the results following the calculation in section 10. Circle the lowest final weight of the two weights that varied between 1.0 mg. This is used as the final weight for calculating TDS. NOTE: If a constant weight is not achieved within three drying cycles, circle the lowest final weight. This is used as the final weight for calculating TDS.

8.3.4 Complete the data package making sure it is properly filled out and the results are highlighted. See sample example. Be sure to include copies of the special instructions.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank is required with every batch of 20 or less samples. The blank is prepared using 100 mL of deionized water. It is to be taken through all steps of the analysis, including filtration. It is not necessary to measure the conductance.

9.1.2 A DCS pair is required with each batch of 20 or less samples. These are to be taken through all steps of the analysis, including filtration. It is not necessary to measure their conductances. DCS are prepared using 100 mLs of standard.

9.1.3 Duplicates may be required for project specific QC.

9.1.4 Spikes may be required for project specific QC. Spikes may be analyzed by mixing equal volumes of DCS and sample. Calculation of the result should be based on the actual volume of sample used in the spike (one half the total volume). The "spiking concentration" is equal to the DCS value.

9.2 Acceptance Criteria

9.2.1 Weight change for blanks must be between -0.5 and 2 mg.

9.2.2 Average DCS recovery must be 90 - 110%.

9.2.3 The RPD for the DCS must be less than 10%.

9.2.5 There are no acceptance criteria for project specific QC.

9.3 Corrective Action Required

9.3.1 Follow the corrective actions outlined in the current Enseco QAPP Manual.

9.3.2 Poor results for the DCS's is usually due to contamination. Be sure all glassware and the filtration apparatus is clean and rerun the samples.

9.3.3 High blanks are also the result of contamination. Low blanks indicate that the evaporating dishes were not completely dry when the initial weight was measured. Rerun the samples.

9.3.4 If the TDS/COND ratio falls outside the limits (0.55-0.81) the sample should probably be rerun for conductance and then for TDS. Consult your supervisor.

10. Calculations

10.1 Calculate blank residue weight:

BLANK = (W2 - W1) X 1000

BLANK = Blank residue weight, mg
W2 = Final blank weight, g
W1 = Initial blank weight, g

10.2 Calculate samples and DCS's:

RESIDUE = (W2 - W1) X 1000

TDS = (RESIDUE) X 1000 / VOL

RESIDUE = Sample residue weight, mg
W2 = Final weight, g
W1 = Initial weight, g
TDS = TDS result, mg/L
VOL = Sample Volume, mL

10.3 Calculate the reporting limit

Rep. Limit (mg/L) = Residue (mg) X 1000 / Vol. (mL)

This is to be done only if the blank is greater than 1 mg or if a sample volume less than 100 mLs was used. Otherwise, the reporting limit is 10 mg/L.

If the blank is between 1 and 2 mg, the reporting limited is based on the blank residue weight. In all other cases, use 1 mg.

The reporting limit is based on either 100 mL **OR** the actual volume used, whichever is lower.

10.4 Calculate the TDS/COND ratio.

TDS/COND ratio = Measured TDS / Measured Conductivity

11. Reporting Requirements

11.1 Units

Report all results in mg/L

11.2 Limits

Results below the reporting limit are reported as ND.

11.3 Significant Figures

Significant figures to report are:

Range	Significant Figures
<10	2
≥10	3

DCS's are reported to three significant figures

11.4 LIMS Data Entry

Since drying times will vary, the analysis date reported will be the date on which the samples were filtered. The holding time is calculated from this analysis date.

11.5 Anomalies

Document any non-standard procedure or any anomalies. Some common anomalies are:

1. Insufficient sample. Reporting limits changed accordingly.

2. Filtrate is turbid.

3. TDS/COND ratio is outside the limits. TDS & COND were rerun and confirmed. The sample contains high concentration of TDS or may contain nonionic species. The conductivity is greater than 10,000 or less than 50 umho/cm so the rule does not apply.

4. Holding time missed.

5. Samples are visibly oily.

See the attached example benchsheet

12. Review Requirements

12.1 All data packages must be complete. They must be properly signed and dated. See example benchsheet.

12.2 Confirm that the special instructions were appropriately observed.

12.3 Check all sample calculations.

12.4 Check the reporting limits.

12.5 Verify that all anomalies are documented.

12.6 The TDS/COND ratio should fall between 0.55 - 0.81. This ratio may not hold for nonionic species or samples high in TDS. It also may not hold for samples with conductances greater than 10,000 or less than 50 umho/cm. Anomalize results that do not fall within these limits.

13. References

13.1 Source Method: EPA Method 160.1

13.2 Related Documents

13.2.1 Standard Methods "Total Dissolved Solids Dried at 180°C", 17th Edition, 1989.

13.2.2 Enseco QAPP

13.2.3 M-EQA-0002 Internal QC Checks--Laboratory Performance QC

13.2.4 LM-RMA-1071: pH, Specific Conductance, and Alkalinity: Automatic Titrator

13.2.5 LM-RMA-1062: Total Suspended Solids (TSS)

13.3 Deviations from Source Method and Rationale

13.3.1 A 103° oven may be used for the evaporation of the sample to dryness.

13.3.2 Constant weight, as defined by the Inorganic Technical Committee of Enseco, is a weight change of 1.0 mg between weighings.

13.3.3 Samples will not be taken through the drying, cooling, weighing cycle more than three times. This is a policy approved by the Enseco Inorganic Technical Committee.

13.4 Updates to SOP (Revision 3.0 to Revision 4.0)

13.4.1 Provision for evaporation of samples on steam bath included.

13.4.2 Maximum number of drying cycles stipulated.

13.4.3 Constant weight defined as weight change of 1.0 mg.

pH, Specific Conductance, and Alkalinity (Automatic Titrator)

SOP No.:	Revision No.:	Effective Date:
LM-RMA-1071	1.0	August 8, 1990

Supersedes: Original

1. Scope and Application

1.1 Analytes

This method is to be used for the determination of pH, Specific Conductance, and Alkalinity. The different forms of alkalinity (total, bicarbonate, carbonate, and hydroxide) can also be calculated.

1.2 Applicable Matrices

This method is applicable to drinking, surface and saline waters, domestic and industrial wastes.

1.3 Reporting Limits

The Enseco reporting limits are:
pH -- Detection limit not applicable
Specific conductance -- 1 umho/cm
Alkalinity -- 5 mg/L as Calcium Carbonate

1.4 Dynamic Range

This method is applicable to all ranges likely to be encountered. As a practical matter, samples with alkalinities greater than about 1200 mg/L as Calcium Carbonate require a smaller than normal volume or stronger titrant in order to keep the titrant volume to a reasonable amount.

Prepared by:	Date:
Lindsay Breyer/Roxanne Sullivan	5/24/88
Management Approval: [signature]	Date: 8/6/90
QA Officer Approval: Tina Rigin	Date: 8/6/90

1.5 Analysis Time

The approximate analytical time is five minutes per sample. Samples with high alkalinity or undissolved solids may require longer titration times.

2. Method Summary

2.1 Samples are analyzed on an automatic titrator.

2.2 The Specific Conductance is measured first by application of a voltage across the conductivity cell in series with a known reference resistance. The ratio of voltages across the cell and the known resistance is measured which allows the resistance of the cell to be calculated. This is then converted to the conductance of the cell. By calibrating against a solution of known conductance, the cell constant is determined and also the specific conductance of the sample in the cell. A thermocouple measures the temperature of the sample and electronically corrects the measurement to 25°C.

2.3 The pH is then determined electrometrically with a glass electrode in combination with a reference electrode. The special glass used in the electrode develops a voltage across it that depends on the pH of the solution being analyzed. The voltage is measured and converted to pH by calibration against buffers of known pH.

2.4 Alkalinity is determined by titrating the sample with a standardized acid to specified endpoints (pH 8.3 and 4.5). Alkalinity is calculated from the volume of acid required to reach the endpoints and is traditionally reported as Calcium Carbonate.

3. Comments

3.1 Interferences

3.1.1 Sodium can interfere with pH determinations at pH's higher than 10. This is controlled by using a "low sodium error" electrode.

3.1.2 Oil and particulate matter in the samples may coat the pH electrode and conductivity cell causing erroneous readings. This will also affect the alkalinity results.

3.1.3 Temperature variations will affect both pH and conductance measurements. The conductance measurements are corrected for temperature automatically. The effect on pH is controlled by performing the measurements at the same (i.e. room) temperature. All samples must be within 2°C.

3.1.4 Salts of weak organic and inorganic acids will contribute to alkalinity. If the alkalinity is intended to be a measure of carbonate and biocarbonate only, the presence of these substances will cause high results.

3.1.5 The alkalinity is titrated to pH 4.5. This endpoint may not be appropriate for all samples.

3.1.6 Specific conductance is extremely sensitive to contamination. The titration head, electrodes, and all glassware must be thoroughly rinsed with deionized water to remove all traces of salts.

3.1.7 Samples not in equilibrium with the atmosphere may exhibit changes in pH and in the distribution of the various forms of alkalinity when exposed to the atmosphere. The sample containers should be filled completely and kept closed until just prior to the analysis. The analysis should be performed as soon as possible.

3.1.8 The pH electrode may exhibit slow or noisy response with high purity waters due to the lack of ionic strength.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco-RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.

4.2 Normal laboratory safety precautions should be followed. There are no special concerns with this method.

5. Sample Collection, Preservation, and Holding Times

5.1 Samples are to be collected in plastic bottles and refrigerated to 4°C.

5.2 Samples for alkalinity must not be filtered, diluted, concentrated, or altered in any way.

5.3 If the conductance measurement cannot be performed within 24 hours of collection, the sample should be filtered.

5.4 The holding time for these parameters will be considered to be 24 hours since all are analyzed at once. The official holding times are:

pH -- 24 hours
Conductance -- 28 days
Alkalinity -- 14 days

6. Apparatus

6.1 Radiometer Automatic Titration System, consisting of autosampler, burette, pH meter and electrodes, conductance meter and cell, temperature probe, stirrer, printer, etc.

6.2 Plastic beakers to fit autosampler.
These must be thoroughly rinsed to remove all traces of salt.

6.3 Oxford pipette calibrated to 10.0 mL, and disposable tips.

6.4 Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

7.1 pH Buffers, 4.0, 7.0, and 10.0
Obtained commercially

7.2 0.01 M Potassium Chloride (Conductance Standard)

Keep a beaker full of KCl in a 105°C oven for routine use. Dissolve 0.7456 g in deionized water and dilute to 1 liter. This solution has a conductance of 1417 umho/cm at 25°C. Prepare monthly.

7.3 0.0500 N Sodium Carbonate

Dry anhydrous sodium carbonate at 250°C for four hours and cool in a desiccator. Dissolve 2.6497 g in deionized water and dilute to 1 liter.

7.4 0.1 N Sulfuric Acid (Strong Alkalinity Titrant)

Very carefully add 3.0 mL concentrated sulfuric acid to approximately 500 mL deionized water and dilute to 1 liter. This solution must be standardized using the procedure given in section 8. Record standardization data and obtain a verification number from the Standards Prep Department.

7.5 0.02 N Sulfuric Acid (Alkalinity Titrant)

Very carefully add 3.0 mL concentrated sulfuric acid to 5 liters of deionized water. Standardize using the procedure given in section 8. Record standardization data and obtain a verification number from the Standards Prep Department.

7.6 Saturated NaCl

Dissolve 35.7 g of NaCl in 100 mL of cold DI.

8. Procedure

8.1 Preparation

8.1.1 Thoroughly homogenize samples before taking an aliquot for analysis. Allow aliquots to equilibrate to room temperature before analyzing.

8.2 Instrument Set-up and Calibration

8.1.2 Standardization of Alkalinity Titrant

Accurately measure 0.05 N Sodium Carbonate into a 250 mL beaker. Use 40 mL if 0.1 N Sulfuric Acid is being standardized or 15 mL for 0.02 N Sulfuric Acid. Add deionized water to bring the total volume to 100 mL.

Titrate with the appropriate strength acid to a pH of about 5. Lift electrode and rinse into beaker. Cover with a watch glass and boil solution gently for 3 - 5 minutes. Cool to room temperature, rinse watch glass into beaker, and continue the titration to the pH inflection point. Calculate the acid normality using the formula in section 10.

Perform all titrations for standardization in duplicate.

8.2.2 Calibration for pH

Be sure that the reference electrode has been filled with saturated potassium chloride and that all electrodes and the titration head are clean and dry (only use Kim wipes to dry the electrodes).

Pour approximately 20 mL pH 7 buffer into a beaker and lower the electrodes into it. Place the titrator in pH mode and press the CAL button when the reading has stabilized. Step through the calibration routine by pressing the CAL button. When Buffer 2 is displayed, rinse the electrodes and immerse in pH 4 buffer. Allow the reading to stabilize and continue with the calibration routine. At each step, make sure that the displayed parameter is correct (e.g. buffer value, temperature, etc.). If you need to change an entry, press the SHIFT button and enter the correct value. When Buffer 3 is displayed, press the pH button to exit the calibration routine (this part of the calibration routine is for a different application not used here). Rinse the electrodes.

8.2.3 Calibration for Conductance

Be sure that the electrodes and titration head are clean and dry (only use Kim wipes to dry the electrodes).

Pipette 20 mL Conductance Standard (0.01 M potassium chloride) into a clean beaker and immerse the electrodes. Verify that the reference temperature is set to 25°C and

the temperature compensation is 2% per degree C. Then adjust the cell constant to give a reading of 1.42 mS/cm. Rinse the electrodes when done. Since the accuracy of this measurement is very dependent upon electrode position it might be advised to calibrate on the first two cups of an autotitrator run. The position of the arms and electrodes are consistent during a run. Calibration should be done daily.

8.3 Analysis

8.3.1 Obtain samples from sample receiving. Check each sample for special instructions. Make copies to put into your data package. Fill out the benchsheet.

8.3.2 Turn on the power to the burette and autosampler. Be sure the titrant reservoir is full. The 0.02 N Sulfuric Acid should be used. If new titrant has been prepared, rinse out the reservoir with it, refill, and flush out the burette and titrant delivery line. Be sure that there are no bubbles in the line.

8.3.3 Set the autosampler to 2 rinses and a delay of 0.4 minutes. Fill two beakers with deionized water (20 - 25 mL) and place them in positions 19 and 20. These are the rinse beakers.

8.3.4 Set the titrator to EP mode and be sure that Method 1 is displayed.

8.3.5 Pipette 20 mL of each sample into a clean beaker and place on the turntable. A 10 mL Oxford pipette may be used to dispense two 10 mL aliquots of sample, if it has been calibrated previously to 10 mL. Record the sample ID and position on the titrator. Allow sample aliquots to equilibrate to room temperature.

8.3.6 Press START NEW CYCLE on the autosampler and the analysis will commence. Results will be automatically printed out and the sampler will stop when all samples have been titrated. At this point, additional samples can be placed on the titrator and analyzed starting at step 8.3.4. Be sure to keep the titrant reservoir filled and replace the water in the rinse beakers periodically.

8.3.7 The titrator has been programmed to deliver a maximum volume of 25 mL titrant. Samples requiring more than this should be reanalyzed using a smaller aliquot or strong titrant (0.1 N Sulfuric Acid). See your supervisor for the corrections to be made in the program for titrator.

8.3.8 Samples with a conductance greater than 50,000 umho/cm need to be run on the high curve. See Appendix 1.0.

8.4 Conclusion

8.4.1 Clean up the work area.

8.4.2 Return samples to Sample Receiving.

8.4.3 Calculate the results according to the calculation section below.

8.4.4 Complete the data package making sure it is filled out properly and all the results are highlighted. Be sure to include the tape and copies of the special instructions. Submit the data package to your supervisor for approval.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 Deionized water and tap water are to be analyzed at least once daily. The conductance of these is to be recorded in the notebook provided for that purpose.

9.1.2 Check a 10.0 pH standard with each new calibration.

9.1.3 One blank and DCS samples must be analyzed in every batch of 20 or less samples. The blank is deionized water.

9.1.4 A blank and standard check are to be performed after every 10 or less readings and at the end of the run. Use the DCS for standard checks. The blank is deionized water.

9.1.5 Duplicates may be required as project specific QC.

9.1.6 Spikes for total alkalinity may be required for project specific QC. Spikes are made by adding 1.0 mL 0.05 N Sodium Carbonate to 20 mL sample. The spiking concentration is 2500 x Normality of Sodium Carbonate.

It is not possible to spike pH, conductance, or the individual forms of alkalinity.

9.2 Acceptance Criteria

9.2.1 Deionized water should have a conductance of 2 umho/cm or less. The alkalinity should be less than 5 mg/L. The pH is usually 5 - 6.

9.2.2 There are no acceptance criteria for tap water. Unusual readings should be reported to your supervisor.

9.2.3 pH 10.0 standard should read within ± 2% of 10.0.

9.2.4 Control limits for accuracy and precision (RPD) on DCS samples are:

Parameter	Accuracy	Precision
pH	98 - 102%	5%
Conductance	95 - 105%	5%
Alkalinity	90 - 110%	10%

9.2.5 Standard check should be within 10% of the expected value.

9.2.6 Blanks should be less than the 2x reporting limits for conductance and alkalinity pH blanks should be around 5 to 6.

9.2.7 There are no acceptance criteria for project specific QC.

9.3 Corrective Action Required

9.3.1 Follow the corrective actions outlined in the current Enseco QAPP manual.

9.3.2 If the deionized water has a conductance more than 2 umho/cm, repeat the reading. If it is still high, the deionized water system may need maintenance. Notify your supervisor.

9.3.3 If the pH 10.0 standards is not within limits, recalibrate and check the standard again. If it is still out of limits, see your supervisor.

9.3.4 If the control limits for the DCS samples are exceeded, the problem must be corrected and the samples reanalyzed. Check the calibration procedures and thoroughly clean the electrodes, titration head, and beakers. The most common problem is contamination, which causes high conductance readings.

10. Calculations

10.1 Standardization of Alkalinity Titrant

NACID = NBASE X VBASE / VACID

NACID = Normality of titrant
NBASE = Normality of Sodium Carbonate
VBASE = Volume of Sodium Carbonate titrated, mL
VACID = Volume of titrant needed for titration, mL

10.2 pH values are recorded directly from the titrator printout.

10.3 Conductance values are taken as-is from the print out. If they are printed out in mS/cm, they must be multiplied by 1000. The units uS/cm are equivalent to umho/cm.

10.4 Three values for alkalinity will be printed if the pH is greater than 8.3. Record the values at pH 8.3 and 4.5 on the bench sheet. The third value is the difference and is ignored. Note that some results will be printed in scientific notation; be careful to check this when recording results.

If the pH is less than 8.3, only one value will be printed (at pH 4.5). Record this value on the bench sheet. P alkalinity is ND on these samples.

Calculate the individual forms of alkalinity:

T = Total Alkalinity = Alkalinity at pH 4.5
P = P Alkalinity = Alkalinity at pH 8.3

	Hydroxide Alkalinity	Carbonate Alkalinity	Bicarbonate Alkalinity
P = ND	ND	ND	T
P < T/2	ND	2 x P	T - 2xP
P = T/2	ND	T	ND
P > T/2	2xP - T	2(T - P)	ND
P = T	T	ND	ND

Record the values for Total, Bicarbonate, Carbonate, and Hydroxide Alkalinity.

11. Reporting Requirements

11.1 Units

11.1.1 pH has units of "units".

11.1.2 Conductance is reported as umho/cm at 25°C.

11.1.3 All forms of alkalinity are reported in mg/L as Calcium Carbonate.

11.2 Limits

11.2.1 All values of pH are reported as measured.

11.2.2 Conductance results less than 1 umho/cm are reported as ND.

11.2.3 Alkalinity results less than 5 mg/L are reported as ND.

11.2.4 All reporting limits must be multiplied by any prep or analytical dilution.

11.3 Significant Figures

11.3.1 pH is reported to one decimal place.

11.3.2 Significant figures:

Result	Significant Figure
<10	2
10	3
>10	3

11.4 LDMS data entry

11.4.1 The usual standards for data entry apply.

11.5 Anomalies

Document any non-standard procedures or anomalies.

Some common anomalies are:

1. Missed holding time.
2. Wrong bottle type used.

12. Review Requirements

12.1 All data packages must be complete. They must be properly signed and dated. See attached example.

12.2 Confirm that special instructions were appropriately observed.

12.3 Check the calculations. Make sure all dilution factors were properly applied.

12.4 Check the reporting limits. Remember that the reporting limits must be multiplied by any dilution factor.

12.5 Make sure all anomalies are documented.

12.6 TDS/COND ratio should fall between 0.55 and 0.81.

13. References

13.1 Source Method

13.1.1 pH is EPA Method 150.1.

13.1.2 Conductance is EPA Method 120.1.

13.1.3 Alkalinity is EPA Method 310.1.

13.2 Related Documents

13.2.1 Additional information was obtained from Standard Method #403 for the alkalinity procedure.

13.2.2 Enseco QAPP.

13.2.3 M-EQA-0002 Internal QC Checks--Laboratory Performance QC.

13.2.4 LM-RMA-1003 Alkalinity (Manual Titration)

13.2.5 LM-RMA-1047 pH (soils and wastes)

13.3 Deviations from Source Methods and Rationale

13.3.1 Successive aliquots of sample are not analyzed for pH as in the reference methods.

13.3.2 Temperature is not reported with the pH measurement. All measurements are made at room temperature (approximately 22°C).

13.3.3 Alkalinity titrations are not performed at 25°C as specified in the reference method.

13.3.4 Method 310.1 contains no provisions for determining the different forms of alkalinity. This part of the method was obtained from Standards Methods for the Examination of Water and Wastewater, 14th Edition.

13.3.5 A fixed endpoint of 4.5 is used for all alkalinity titrations.

13.4 Updates to SOP (Original to Version 1.0)

1. Format updated.
2. Significant figures changed.
3. Appendix 1.0 added for high conductance curve.
4. pH 10.0 standard will be checked with each calibration curve.

Appendix 1.0 High Conductivity Curve

1. Calibrate on saturated NaCl. The cell constant should be adjusted so the conductivity of the NaCl reads 251,000 umho/cm.

2. There is no QC.

3. Run your samples. Run DI rinses between each sample and have a double rinse cycle.

pH, CONDUCTANCE, ALKALINITY

ANALYST RS
I E/LOT 06AUG90 R
S. KE —

CONTROL (8812 / _____): pH 9.0 / _____
COND 1130 / _____
TITRANT: 0.025 N ALK 200 / _____

BKR #	SAMPLE #	VOL ml	pH	COND µS	ALKALINITIES, mg/L @8.3	@4.5	Hydr	Carb	Bicarb	Total
1	TAP		5.7	1.26	—	4.073	ND	ND	ND	ND
2	DI		5.4	150.	—	3.548	ND	ND	ND	ND
3	pH 10		10.01							
4	Blank		5.6	1.8	—	3.548	ND	ND	ND	ND
5	DCS		8.97	1134	125.6	204.5	46.7	157.8	ND	204
6	DCS		8.98	1135	128.6	209.8	47.4	162.4	ND	210
7	ABCD-01		8.9		125.6	204.5	46.7	157.8	ND	204
8	02		7.6	1520	—	49.79	ND	ND	49.8	49.8
9	ABCD1-01		4.5							
10	-02		6.2	7.0						
11	Blank		5.4	1.5	—	3.421	ND	ND	ND	ND
12	Standard		8.97	1135	125.6	204.5	46.7	157.8	ND	204

12. PERFORMANCE AND SYSTEM AUDITS

Enseco laboratories participate in a variety of federal and state programs, (including the U.S. EPA CLP), that subject each of the laboratories to stringent system and performance audits on a regular basis. A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff and procedures in place to generate acceptable data. A performance audit verifies the ability of the laboratory to correctly identify and quantitate compounds in blind check samples submitted by the auditing agency. The purpose of these audits is to identify those laboratories that are capable of generating scientifically sound data. Enseco is certified to perform environmental analyses under programs administered by the U.S. Department of Energy, U.S. Air Force, U.S. Navy, and over 20 states. The most current list of Enseco certifications is available upon request.

In addition to external audits conducted by certifying agencies or clients, Enseco regularly conducts the following internal audits:

- Periodic (at least yearly) audits conducted by the Corporate QA Office.
- Special audits by the Divisional QA Director or Corporate QA Office when a problem is suspected.

Enseco laboratories also routinely analyze check samples as described below:

- Laboratory Control Samples (DCS, SCS, and method blanks) are analyzed at a frequency equal to at least 10% of the total number of samples analyzed (see Section 11).
- All Enseco laboratories participate in the analyses of EPA check samples provided under the Water Supply (WS) and Water Pollution (WP) Performance Evaluation Studies. The results of these PE samples are tabulated by the Corporate QA Office to identify performance trends within the Enseco laboratories.

- The majority of the Enseco laboratories are CLP labs and thus analyze organic and/or inorganic CLP PE samples on a quarterly basis. The results of these analyses are also tabulated and evaluated by the Corporate QA Office.

- The laboratories participate in multiple state certification programs (including New York, New Jersey and California) which require that PE samples be analyzed periodically.

The results of these check samples are used to identify areas where additional training is needed or clarification of procedures is required.

Enseco-RMAL has been audited by the Central Regional Laboratory in Region V.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
230 SOUTH DEARBORN ST.
CHICAGO, ILLINOIS 60604

FACSIMILE REQUEST AND COVER SHEET

OFFICE/PHONE (303) 421-6611 (Phone) (303) 431-7171 (FAX)	MACHINE NO. / VERIFICATION NO.
TO: Lindsay Breyer, Jerry Parr	ENSECO/RMP[redacted]
FROM: David A Payne	
PHONE NO. (312) 886-1970	MAIL CODE 5SCRL
OFFICE LSSS, CRL	
DATE 5-31-90	NUMBER OF PAGES, INCL[redacted] SHEET 12

PLEASE NUMBER ALL PAGES

INFORMATION FOR SENDING FACSIMILE MESSAGES

EQUIPMENT	FACSIMILE NUMBER	VERIF[redacted] NO.
PANAFAX UF-640	FTS-886-2591 COMM: (312) 886-2591	353-[redacted]

ENVIRONMENTAL SCIENCES DIVISION

WILLIAM H. SANDERS III, DIRECTOR

PAGE 1 OF 12 PAGES

unofficial
Here is a copy of 4 County Landfill. The [redacted] site is the same report except for site specific i[redacted] bottom of p 2 and on p. 3 of cover memo. Attached memo will be official when WWE provides it to us.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
230 SOUTH DEARBORN ST.
CHICAGO, ILLINOIS 60604

MAY 0 9 1996

REPLY TO THE ATTENTION OF:
5SCRL

MEMORANDUM

SUBJECT: Labortory Evaluation of ENSECO/RMAL, Arvada, Colorado for the RFI at the Four County Landfill, Fulton County, IN

FROM: Curtis Ross, Director Chuck Elly for
Central Regional Laboratory

TO: William E. Muno, Chief
RCRA Enforcement Branch

ATTN: Jonathan Adenuga

Attached is the final laboratory evaluation report for ENSECO/Rocky Mountain Analytical Laboratory (RMAL), Arvada, Colorado for Appendix IX determinations.

ENSECO/RMAL is appropriate and acceptable for Appendix IX determinations, with correction of the technical deficiencies listed below. This list is relatively small in size for the large number of analyses to be done. This laboratory evaluation report does not discuss dioxin/dibenzofuran testing to be done at ENSECO/CAL using Method 8280.

1. Changes in the detectability of specific organic compounds are suggested, including 2 of 9 phosphorus containing pesticides when determined by Method 8270.

2. Reference 5 Point Calibration Curves should be available for all compounds determined by gas chromatography (GC).

3. Acceptance limits for DDT/endrin breakdown checks should be brought into line with specifications of Method 8080.

4. Method 8150 for determining herbicides in soils has severe performance problems. The SW-846 methodology itself provides conditions for producing aldol condensation products that interfere in the chromatography of analytes. ENSECO/RMAL in-house SOP for herbicides needs definite improvements for both waters and soils. ENSECO/RMAL has agreed to select the best available modifications of the methodology to provide the best quality data during the time available before RFI sampling is initiated. Method 8150 for soils must be discussed on a case-by-case basis for each RFI site.

5. RFI QAPPs should reflect the manual distillation colorimetric procedure used for sulfide in soils or waters. RCRA Method 9030 is not in use for sulfide at ENSECO/RMAL.

The attached report recommends strongly that sample extract clean-ups be practiced for soils (and waters when appropriate) so as not to dilute interferences, surrogates, and matrix spike compounds with corresponding increases in reporting limits. ENSECO/RMAL is currently re-evaluating their extract clean-up procedures for Methods 8080, 8140 and 8150. QAPP descriptions of analytical methods need to define extract clean-ups to be used in addition to sample extraction procedures.

The report recommends additional surrogate compounds for Method 8080 finalization of a surrogate for Method 8150, and additional matrix spike compounds (phosphorus pesticides) for Method 8270.

Weaknesses in ENSECO in-house SOPs are noted for inorganics and Method 8150. Other organic SOPs are out-of-date and need to be updated.

The attached report discusses ENSECO/RMAL reporting limits, specifically for water-miscible volatiles, and 9 phosphorus pesticides and other semi-volatiles by Method 8270. Reporting limits for Methods 8080, 8140, and 8150 are discussed in relation to their low standard concentration of a 5 Point Calibration Curve. We realize reporting limits are a dynamic process for any laboratory. Recommendations are made for reporting compounds when identified present at less than the normal reporting limit. ENSECO/RMAL is asked to review each QAPP for accuracy of reporting limits for the 3 GC Methods.

The attached report recommends strongly that ENSECO'S in-house controls (LCS, DCS, SCS) not replace the accuracy objectives tied to QAPP and site specific matrix spike and surrogate spike recoveries. Sample extract clean-ups are also tied to this issue so that spike compounds will not be diluted to non-detectable levels.

Comments are made for site-specific items of the Four County Landfill RFI

1. The ENSECO/RMAL QAPP, dated Decmeber 28, 1989, does not address test procedures and QC practices for non-appendix IX determinations of TOC, TOX and phenols, etc.

2. Any phenolics testing should define sample distillation and colorimetric procedures. The need for the chloroform extraction option of the phenolics colorimetric test needs to be discussed if this parameters is measured.

3. The lab evaluation by the CRL of ENSECO has not addressed air monitoring at the subject site.

4. The reporting limit for methylene chloride appears unreasonably large.

cc: D. Payne
G. Schupp
E. Evangelista
K. Gunter

5SCRL:DPayne:dlw:5/10/90:A:\DAVE\RMAL.RFI
CHRON
FILE

I. INTRODUCTION

During January and February 1990, the Region V Central Regional Laboratory (CRL) reviewed analysis records (Appendix IX) for 1 water (field blank), 2 soils, and 2 wastes as provided by ENSECO/RMAL. These were reviewed for conformance with ENSECO SOP's (or test procedure descriptions) dated February 1989 and ENSECO QC practices, for conformance with SW46, 3rd ed., requirements, and for technical merit. An initial draft lab evaluation report was provided ENSECO/RMAL February 14, 1990 based on analysis record. ENSECO/RMAL and the Region V CRL have resolved most differences concerning Appendix IX determinations. A final laboratory evaluation of ENSECO./RMAL is provided below for RCRA Appendix IX determinations. This memo report does not address any dioxin/dibenzofuran analyses by Method 8280 to be done by ENSECO/RMAL.

II. APPENDIX IX COMPOUNDS OR PARAMETERS

A. The following compounds should be noted as not being detectable by ENSECO/RMAL using GM/MS methods.

1. 4-Nitroquinoline-1-oxide
2. p-Phenylenediamine
3. Hexachlorophene
4. Pyridine (soils)
5. 1,4 Dioxane at a D.L. of 100 ug/l in water. 1,000 ug/l standards of 1,4 Dioxane did not provide sufficient sensitivity to warrant using a D.L. of 100 ug/l.

6. Ethyl Cyanide at a detection limit of 5.0 ug/l. The first 4 compounds are a fact of life. Many labs can not detect these compound. There is disagreement between the CRL and a RMAL on D.L. for 1,4-Dioxane. The ethyl cyanide was not discussed previously with RMAL.

7. Dimethoate and Famphur

The first 4 compounds are a fact of life. Many labs can not detect these compounds. The detection limit for 1,4 dioxane should be increased. There is disagreement between RMAL and the CRL for a reporting limit of 5.0 ug/l for ethyl cyanide. The CRL believes it should be larger (50 ug/l or more) as is common for other waste miscible volatile compounds.

B. The following compounds should be added to data reports for a RFI.

1. Cis-1,2-dichloroethene (presently part of RMAL Method 8240 calibration standards)

2. α & γ chlordane (optional) - this is presently part of RMAL Method 8080 calibration standards and is a more meaningful measurement than technical chlordane.

C. Each RFI must determine whether Method 8140 is to be used to determine a phosphorus containing pesticides (at high sensitivity) or if the 9

pesticides can be determined by Method 8270 (at low sensitivity) for more cost-effective analyses (elimination of a tast procedure). There is a question whether the high initial pH value of the RMAL continuous liquid-liquid extraction of waters will destroy any of the 9 pesticides. Method 8140 extractions are done at a neutral pH value. The choice of the methods for the 9 phosphorus pesticides is based on project objectives and intended data usage(s). If the 9 pesticides are to be done by Method 8270 the following problems are encountered:

1. Dimethoate is destroyed at high pH values and will not be detected by BNA extractions of waters. This was confirmed by ENSECO/RMAL on May 1, 1990 and is documented by EPA method validation studies for Appendix IX.

2. The data review by the CRL found Famphur to have zero mass spectral response.

3. On May 1, 1990, RMAL provide data to support acceptable recoveries for the remaining compounds for BNA extractions from waters.

D. N-nitrosodiphenylamine and diphenylamine can not be differentiated (in the same way as m-cresol and p-cresol can not be differentiated) by Method 8270. These compounds should be noted as "either/or" in final data reports.

III. GC/MS INSTRUMENT OPERATION - METHODS 8240 AND 8270

The data package reviewed provided examples of excellent GC/MS operations. Procedures in effect follow and are consistent with the technical requirements of the CLP Statement of work. ENSECO-RMAL was the first lab reviewed for RCRA programs where no errors have been observed for these operations. Appendix IX compounds have been properly apportioned or selected for the 2 methods, however, each Region V RFI will need to determine whether 8140 should be used for the 9 pesticides, or whether Method 8270 will be appropriate. This is a site-by-site decision.

Calibration of the 2 Methods and instruments are done using a 5 Point Calibration Curve for reference for the CLP SOW TCL compounds. Each day after initial MS tuning and blanks, two standards are used to update the instrument response factors and retention times using authentic standards. One standard contains the CLP TCL compounds and one the extra Appendix IX compounds (a 5 Point calibration Curve is not available for the extra Appendix IX compounds, but this has been acceptable to Region V for any "extra" Appendix IX compounds that are not site-specific contaminants).

Surrogate recoveries were available for Method 8240 because they were added after extraction of the wastes with methanol. Both surrogates showed little, if any, deviation from 100% recovery. Internal standards response was relatively constant for both Methods 8240 and 8270 and of excellent quality. The in-house Lab control Standards and Surrogate control Standards for both soils and waters did not significantly differ from 100% recoveries. These are

indicative of excellent instrument operation and calibration for Appendix IX compounds.

The one difference in instrument operation between EPA programs and ENSECO's industrial clients is that tuning checks and continuing calibration standards are not determined every 12 hours for industrial clients as they are one for EPA programs.

Any questions we have had concerning preparations of calibration standards (proprietary in nature) for Methods 8240 and 8270 have been resolved by ENSECO/RMAL. Standards from authentic compounds are used at least daily to update GC/MS libraries for these 2 methods.

We do have questions on use of Reporting Limits or "PQL's" used by ENSECO. For 9 water miscible volatiles, the reporting limits appear inconsistent with each other. In two cases, acrolein and acrylonitrile reporting limits are larger than the daily standard used for instrument calibration. Reporting limits for aramite, isosafrole, and symmetrical trinitrobenzene appear too small. The reporting limits reported by RMAL for 7 phosphorus pesticides on February 14 1990 appear too large (50 or 100 ug/l) for their response factors by Method 8270.

Review of the 1989 data package showed RMAL to report the presence and concentration of all MS "hits" even if the concentration was less than the reporting limit. Data reduction would then change these values to "less that reporting limit even though acceptable mass spectra are present (The CLP will report these low concentrations with a "J" qualifier).

Recommendations:

1. Report all positive MS "hits" with acceptable mass spectra as recommended by CH2M-Hill in their letter of March 2, 1990.

2. Justify the large reporting limits for 7 of the 9 Method 8140 pesticides. Whey don't they approximate 10 ug/l?

3. Significantly increase the PQL for 1,4 dioxane and Ethyl Cyanide.

4. Justify why Daily Std concentrations are lower than their reporting limits for Acrolein, and Acrylonitrile. How can you calibrate or update an instrument using concentrations smaller than their reporting limits?

5. Is there a rationale for selection of reporting limits for the water soluble compounds of Method 8240?

6. Why is the reporting limit set at 10 ug/l for sym. trinitrobenzene?

7. Justify whey Famphur is reported at all. RMAL recently provided data to indicate Dimethoate is not detected due to loss in the BNA extraction.

IV. GC INSTRUMENT OPERATIONS - METHODS 8080, 8140 AND 8150

The instrumental operations for ENSECO Methods 8080, 8140, and 8140 are fairly straight forward, after review of a "typical data set". Five point calibrations are provided for the CLP and TCL pesticides of Method 8080. Single standard solutions are injected for chlorobenzilate, isodrin, kepone, and the multicomponent Aroclors, tech. chlordane, and toxaphene. External standards are used to quantify these compounds in sample extracts. ENSECO is to be complimented on their thorough documentation of Rention Time windows and their use in their Nelson Data System for the CLP TCL pesticides.

1. Recommendation:

   ENSECO needs, as a reference, 5 point calibration curves for the determination of linearity, acceptable instrument response, detection limits, etc., for the 3 Appendix IX pesticides, γ-chlordane, α-chlordane, and at least 4 of the routinely determined Aroclors (1242, 1248, 1254, and 1260). We would encourage use of the Nelson Data System for these latter compounds, but do not require it. It is helpful to use the data system to plot the reference calibration curves, instead of reviewing individual calibration factors. ENSECO/RMAL has no problem with calibration curves used a reference so long as single standards can be used on a daily basis.

2. Recommendation:

   ENSECO should consider that either DDT or endrin degradation must be <20% when pesticides are reported. We would encourage these degradations to be smaller. We realize that automated instrument operation does not allow these determination to be made prior to sample analysis; however, SW-846 Method 8080 mandates the degradation be less than 20% for either compound.

The instrument operations for Method 8140 were routine; however, 3 of the 9 compounds were inappropriate concentrations for their 5 point calibration curves. This is documented in case the methodology is used for other projects.

ENSECO's Method 8150 had a 5 point calibration curve, of excellent quality. The SOP specifies the standards are prepared in-house using the methylation reagent that is used for samples. Telephone conferences with ENSECO indicated the standards (for the 3 herbicides of interest) are purchased methyl esters.

3. Recommendation

   SW-846, 3rd edition, and ENSECO's in-house SOP both specify preparation of standards using the in-house methylation reagent - diazomethane. This needs to be resolved. If standards are to be purchased as methyl esters, controls will need to be carried through each set of sample preparations.

4. Observation:

In the "typical" data set for Method 8150, a typical 5 point calibration curve was constructed, using methyl esters. The SOP for Method 8150, specifies a 4 point calibration curve of 3 standards with the origin being the 4th point. No calibration curve was submitted for our evaluation. The Nelson Data System for dual acquisition from 2 columns supplied with the data package gave information as to the consistency of the response factor (RF) through the entire calibration range, except for Dinoseb on channel 1 and DCAA and Dinoseb on Channel 2. RF's for all other compounds were consistent. A calibration curve or consistent RF would be helpful in estimating Reporting Limits for the Herbicides, along with herbicides prepared from the in-house methylation reagent.

5. Statement:

ENSECO is to be complimented for use of their dual (simultaneous) capillary columns for simultaneous confirmation, identification, and quantitation.

V. REPORTING LIMITS FOR METHODS 8080, 8140, AND 8150

A variety of reporting limits or reporting limits were questioned or discussed in previous correspondence for the GC methods. The reporting limits appear arbitrary for ENSECO.

1. Recommendation:

The final reporting limit used should be based on the amount reasonably detected in the sample extract (ug/ml), multiplied by the extract volume (10ml for Method 8080 or 1ml for Method 8270), multiplied by any extract dilution, and related to 1 liter of water or 10 to 30g of soil aliquot. If no interferences are present, the reporting limit should approximate or bear some relation to the lowest concentration value measured in a compound's 5 point calibration curve. During sample analysis for the RFI, reporting limits or sample concentrations identified from 2 columns should be reported through the above calculations.

VI. SAMPLE EXTRACT CLEAN-UPS

For the "typical" (or "non-typical") data set reviewed, interferences in sample extracts of non-typical soils or wastes had a bigger effect on sensitivity of analysis than any other factor. Extracts were not cleaned for Methods 8270 or 8150. Interferences caused dilution of the extract (so as to dilute out surrogates) for Method 8080 even though the extracts were taken through the 50% ether fraction on Florisil. Interferences in sample extracts had a larger negative effect on data quality than any other factor. The excellent instrumentation of ENSECO could not be used to its full capability due to over-dilution of extracts to minimize interferences.

Decisions for surrogate concentrations, matrix spike concentrations, and whether to clean-up an extract appear the responsibility of the sample preparation groups at ENSECO. If clean-ups are not done, but extracts still need to be diluted, or cleaned up, the extracts were not recleaned for the "typical" data set.

1. Recommendation

   If sample extracts are "dirty" or exhibit significant interferences with no clean-up they should be retested after cleaning. This is a non-negotiable issue.

2. Recommendation:

   We would encourage ENSECO to use gel permeation clean-ups of soil extracts,as they do for the CLP, for Method 8270. We can not require it at this time since we do not know the effect of gel permeation on the extra Appendix IX compounds. ENSECO/RMAL can certainly use gel permeation on clean-ups for priority pollutants or CLP SOW TCL compounds.

3. Recommendation:

   The use of 6% ether or 15% ether/hexane elutions of soil or water extracts can easily contain less interferences than the 50% ether/hexane fraction. The use of a 6% ether/hexane fraction will contain most of the chlorinated hydrocarbon pesticides and Arcolors of interest for Method 8080. See initial discussions of February 15, 1990. While we can not argue with "acceptable" results obtained from a combined 6%, 15% and 50% ether fraction, we believe a 6% and 15% fraction can be in order when the combined fractions show or exhibit gross interferences.

   A 6% ether/hexane fraction is necessary if silicic acid chromatography is to be used to separate pesticides from PCB's

4. Recommendation:

   We assume and recommend that ENSECO will clean all soil extracts for Method 8150. Evaluations of other Region V RFI labs have found that Method 8150 extracts of waters and soils definitely need "clean-ups", or elimination of interferences.

5. Recommendation:

   We hope that any technical discussions between ENSECO-RMAL, RFI project managers, and Region V will define reasonable extract clean-ups for the planned RFI. RFI QAPP's need to define the extraction and clean-up procedures to be used for soils and wastes.

7. Recommendations:

   Any data package received by ENSECO should have a prep-log for sample extracts, with clean-ups tied and documented to specific SOP's or procedures.

VII. MATRIX SPIKES AND SURROGATES

Discussions on evaluation of the data set from ENSECO-RMAL centered on appropriate surrogates and matrix spikes. Previous comments were not specific but centered on over-dilution of matrix spikes and surrogates, in sample extracts for soils and wastes.

1. Recommendation:

   Add 2 or 3 of the Method 8140 pesticides (phosphorus) to the matrix spike mixture for Method 8270.

2. Recommendation:

   Consider increasing the matrix spike and surrogate concentrations for Methods 8270, 8080, and 8150 if they are being diluted-out for soil extracts. Use medium conc. spike levels for medium concentration samples dilutions.

3. Recommendation:

   Use tetrachloroxylene and decachlorobiphenyl as surrogates (advisory) for Method 8080. The DBC surrogate is a waste of time to represent florisil clean-ups.

4. Recommendation:

   ENSECO-RMAL needs to finalize its use of either DCAA or 2,4-DB as a surrogate for Method 8150.

5. Recommendation:

   The data resulting from "LCS, "DCS," and "SCS" in-house controls should be part of any data package however, any such reported data should be documented in such a way that the data user can determine whether it is a solid or water type, whether it was prepared in the same lot of samples analyzed, and whether it was tested on the same instrument and same day as the samples of interest.

   We consider these control samples to be of use, but that their acceptance criteria be based on in-house statistical estimates and not on CLP criteria developed from actual samples, taken through all extract clean-ups. These control samples should not replace site-specific surrogates and matrix spikes.

A RFI QAPP should establish a surrogate matrix, spikes and matrix spike/matrix spike duplicate compounds to use.

VIII. ADEQUACY OF SOP's

A. SOP's for ICP Metals (Method 6010, 200.7, or 200.7 CLP-S) and graphite furnace atomic absorption metals analysis do not exist at ENSECO/RMAL for industrial clients. Interelemental correction factors for ICP measurements were never provided to Region V initially requested. Cyanide SOP's have not been available but review of data package shows colorimetry (1 of the options of CLP SOW) that can be easily referenced. Review of metals results indicated test procedures are equivalent to CLP SOW, and that metals analyses are done excellently with technical merit.

B. SOP's for sulfide are not available, Reference to RCRA Method 9030 (iodine titrimetric) is inappropriate. A calorimetric procedure after manual sample distillation is in use at RMAL. A QAPP.

C. The SOP's for organic analyses are out-of-date. Basic GC/MS and GC procedures are unchanged but capillary columns have replaced packed columns. SOP's available for review often did not reflect the sample extraction/clean-up procedures in use for organics. Confusing options are available in order to accommodate for all ENSECO labs. Method 8150 is the worst example.

D. As a general rule, SOP's reviewed were not up-to-date versus the data package reviewed.

IX. METHOD 8150

This method appears not applicable to soils because of an "aldol condensation" product(s) from acetone (used during sample extraction) that destroys, or interferes with, the chromatography of analyses. Interferences can be so severe that the analysis may be a waste of time and money. Part of this is due to a methodology problem of U.S EPA itself. ENSECO will be working to improve the situation for soils; however, we can not determine how successful they will be during the next 2 months. Method 8150 for soils has not been a successful analysis for other RCRA RFI labs evaluated by Region V.

Recommendation: The use of Method 8150 for soils must be discussed on a case-by-case basis for each RFI site.

X. MISCELLANEOUS ITEMS

A. We noted a severe interference for tin during soil and waste analysis by ICP emission spectroscopy. We believe this could be due to an spectral interference from sulfur. We do not know if ENSECO can correct for it, or provide a different type of background correction for tin.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
230 SOUTH DEARBORN ST.
CHICAGO, ILLINOIS 60604

REPLY TO THE ATTENTION OF:

MEMORANDUM

5SCRL

DATE:

SUBJECT: Laboratory Evaluation of Rocky Mountain Analytical, Arvada, CO, of ENSECO, Inc. for the Modern Plating Corp. Site, Freeport, Illinois

FROM: Curtis Ross, Director
Central Regional Laboratory

TO: William E. Muno, Chief
RCRA Enforcement Branch

ATTN: Eileen Helmer

I. INTRODUCTION

David A. Payne, Chemist, visited Rocky Mountain Analytical Laboratory (RMAL) of ENSECO, Arvada, CO to evaluate this laboratory for the RCRA Facility Investigation (RFI) to be done at Modern Plating Corp., Freeport, Illinois. The RFI is to be done by Foth and Van Dyke, Green Bay, Wisconsin. Analytical chemistry measurements to be done by RMAL involve:

1. Approximately 75 groundwater samples to be tested for volatile organic compounds (VOC's) by Methods 601 and 602. The VOC's to be tested are provided by Tables 2-3 and 2-4 of the September 1988 Data Collection QA Plan prepared by Foth and Van Dyke.

2. Approximately 9 water samples and 36 soil or sludge samples to be tested for RCRA Appendix IX contaminants using

    a. Method 8240 for VOC's

    b. Method 8270 for BNA Semi-Volatiles

We have reviewed the ENSECO/RMAL document "Analysis of Appendix IX constituents", February 1988 and find the listed Appendix IX contaminants to be consistent with EPA's list of July 9, 1987, for the stated methods of analysis.

3. Approximately 9 groundwaters to be tested for Appendix IX metals/inorganics. The RCRA Enforcement Branch needs to assure that cyanide will be tested in these samples-either by RMAL or by the Foth and Van Dyke laboratory.

4. A certain number of soils/sludges to be tested for VOC's after TCLP Zero Head Space Sample Preparation.

During the laboratory evaluation, Mr. Payne met with 1) Debbie Fazio, 2) Tony Mariano, 3) Jan Redenbarger, QA Staff, 4) Jeff Laury, Manager of GC/MS, 5) Mike Hoffman, Supr. of VOC GC Analysis, 6) Susan Brillante, Manager of GC Analysis, and 7) Dave Roberts, Supr. of Metals Analysis, and 8) Brian Rahn, Sales Representative, all of RMAL/ENSECO.

II. SUMMARY

1. Rocky Mountain Analytical (RMAL) of ENSECO has the analytical capability necessary for the required Appendix IX measurements at the Modern Plating. Detailed recommendations for changes in RMAL's routine procedures for conformance with the subject sites' QA Plan are provided at the end of the evaluation report details for 1. Methods 8240 and 8270, 2. Metals Analysis, and 3. Methods 601/602. Recommendations are made in the areas of 1. raw data records needed to be part of the data packages sent by RMAL to Foth and Van Dyke, 2. blank correction not being made to sample analysis results, 3. results being reported on a dry weight basis for soils/sludges, 4. project specific QC, 5. frequency of certain QC audits, and 6. RFI work should be done at RMAL and not sent to other ENSECO labs.

raw data pkg
NO blk subtraction
dry wt. basis.

2. Recommendations are made that RMAL Method 601/602 is not acceptable for volatile organic compounds (VOC's) unless confirmatory analyses are done as required by the designated reference methods. Note that Method 601, Method 602, Method 502.1, and Method 503.1 each provide a second, packed gas chromatography column suitable for confirmatory analysis of samples of unknown VOC identity or suggest the use of GC/MS. Confirmatory analysis of VOC identity is part of this EPA purge and trap GC methodology. We are uncertain whether previous VOC results at the Modern Plating site had any confirmatory analyses when using Methods 601 and 602. This includes data previously provided by Foth and Van Dyke for any routine RCRA groundwater monitoring.

RMAL has proposed the use of GC/MS Method 8240 VOC analyses instead of Method 601/602 as being more cost effective than 601/602 used with confirmation by their version of EPA Method 502.2.

Method 8240 will certainly provide unambiguous identity of the target VOC's (with cis-and trans-1, 2-dichloroethene identified and quantified as their total content).

The choice between RMAL Method 601/602 or Method 8240 is now the responsibility of the Region V RCRA Enforcement Branch and Foth and Van Dyke. The following points need to be considered in this choice.

a. Method 8240 provides ENSECO reporting detection limits of 5 or 10 ug/l for each VOC versus 1 or 2 ug/l for each VOC by Method 601/602. The detection limit differences will not be important for "dirty" groundwaters since these samples will have to be diluted into the calibration range of each instrument.

b. Method 8240 will provide unambiguous identification of VOC's and its sensitivity should be sufficient to define movement of VOC contaminants at the site from technical hydrogeology considerations. Method 8240 has the capability to provide tentative identifications of unknown VOC's (or non-target VOC's) if this is needed.

c. The sensitivity of Method 601/602 will be needed to determine compliance with any EPA drinking water standards for synthetic VOC's. The 8 regulated SVOC's have standards between 1 and 5 ug/l for benzene, vinyl chloride, 1, 2-dichlorethane, etc.

d. Table 2-3 of the Modern Plating QA Plan provides VOC's to be tested by Method 8010 or 601. Table 2-4 provides the aromatic VOC's listed by Method 602. Use of either RMAL Method 601/602 or 8240 will cause Table 2-3 and 2-4 to be changed in the following ways.

   i. trans- 1, 2-dichloroethene should be reported as the sum of cis- and trans- 1, 2-dichloroethene by Method 8240.

   ii. Tables 2-3 and 2-4 will be combined into 1 Table of all VOC's to be reported by Method 8240.

iii. Xylenes should be added to Table 2-4 if Method 602 is to be used or to a combined Table for Method 8240 since xylenes are part of RMAL's routine Methods 8240 and 602. Xylenes can well be important site-specific VOC's if aromatic hydrocarbons are present.

iv. The two priority pollutant Freons VOC's should be added to Table 2-3 if RMAL Method 601 is used. These 2 VOC's are not part of RMAL's routine Method 8240 but are part of their extra Appendix IX list for Method 8240.

v. 2-Chloroethyl vinyl ether should be deleted from Table 2-3 or any combined Table since this VOC is no longer part of RMAL's VOC methods because the CLP SOW has deleted it. This VOC has never been noted in any Region V samples. It is very unstable in standards and samples. Modern practices of now preserving VOC samples with HCl to pH <2 will also destroy this VOC. This priority pollutant VOC has never been of any practical significance.

vi. RMAL Method 8240 does not routinely report the 3-dichlorobenzenes of Table 2-3. They are routinely tested by Method 8270. If 8240 is to be used by RMAL the dichlorobenzenes will have to be deleted from the VOC list, or Foth and Van Dyke will have to request a modification of the 8240 methodology at RMAL to require longer retention times. RMAL Method 602 does provide the dichlorobenzenes. The selection of Method 8240 at RMAL may have increased costs if dichlorobenzenes are required for the VOC list.

vii. Table 2-3 and Table 2-4 of the QA Plan will have to be changed regardless whether RMAL uses Method 601/602 or Method 8240. The important, known site specific VOC's at Modern Plating site will be measured by either methodology.

3. ENSECO Inc. will shift work to different laboratory locations if a single laboratory location becomes overloaded. We discussed during the evaluation that RMAL should receive all Modern Plating site samples, so that a laboratory evaluation would not be needed for any other ENSECO laboratory. RMAL was agreeable to performing all lab work at their Arvada, CO location.

III. METHOD 8240-GC/MS FOR VOC's
METHOD 8270-GC/MS FOR SEMIVOLATILE ORGANICS

RMAL uses the U.S. EPA Contract Laboratory Program (CLP) Organics Statement of Work (SOW) procedures for these methods including QC practices. The surrogate compounds (Section 5.3 of Method 8240 and Section 5.5 of Method 8270), internal standards (Section 5.4 of 8240 and Section 5.2 of 8270), matrix spikes, if used, (5 VOC's of Section 5.7 Method 8240 and BNA's of Section 5.6, Method 8270) are used.
Five point calibration curves are used to establish initial calibration conditions (See Section 7.2 of Method 8240 and Section 7.3 of Method 8270).

The calibration curves are deemed acceptable if the appropriate SPCC and CCC calibrations are deemed acceptable (5 SPCC's and 6 CCC's for Method 8270) and are used for reference until a new calibration curve needs to be established for a GC/MS instrument.

The CCC checks are also used to determine that daily instrument calibration, using a single standard solution, does not significantly deviate from the 5 point calibration curve using the CCC's (See Section 7.3.4 of Method 8240 and Section 7.4.4 of Method 8270). The SPCC checks are used to determine that minimum instrument sensitivity is achieved on a daily basis (See Section 7.3.3 of Method 8240 and Section 7.4.3 of Method 8270. Each day of instrument use, the GC/MS tuning standards (Section 7.3.1 of Method 8240 and Section 7.4.1 of Method 8270) are used to demonstrate acceptable MS performance. Throughout the analysis run, surrogate compound recoveries and internal standard response are monitored for acceptability using criteria equivalent to the CLP SOW.

For routine Appendix IX (or RCRA) measurements, RMAL does not measure the GC/MS tuning standards or check the SPCC and CCC responses every 12 hours as specified by the CLP SOW and SW-846, 3rd edition. Routine RCRA measurements depend on the surrogates, internal standards, and other professional MS judgments. If RMAL's clients specify the 12 hour checks are to be done, they will do so. The Modern Plating sites QA Plan dictates use of SW-846, 3rd edition. The 12 hour MS and calibration checks should therefore be done every 12 hours.

To provide GC/MS instrument calibration for the Appendix IX VOC's or semi-volatile compounds not common to the CLP SOW TCL's, a single standard is tested daily, to establish MS and quantitation conditions, using authentic standards compounds. A 5 point calibration curve is not available.

This is common practice for environmental laboratories at this time. The extra Appendix IX compounds are rarely, if ever, detected. Review of the Modern Plating QA Plan indicates known site-specific contaminants are not part of the extra Appendix IX list (compared to the CLP TCL's).

For reporting or detection limits, RMAL uses common detection limits for all of its labs and instruments. These correspond to Appendix C to the current CLP SOW, and to the PQL's of Table 2 of Method 8240 and Table 2 of Method 8270.

RMAL can provide smaller reporting limits for specific instruments; however, standardization of results between instruments in a single lab has dictated the PQL's be used for all results.

Each day of analysis, Lab Control Standards (LCS's) are tested in duplicate after initial GC/MS calibration checks, and a Surrogate Control Standard is tested. These solutions are carried through sample preparation steps, but are prepared in reagent water. The LCS utilize the 5 matrix spike VOC's and 11 matrix spike semi-volatile compounds of the CLP SOW for Methods 8240 and 8270 respectively. Surrogate Control Standard (SCS) results are reported for each set of samples extracted and tested, and contain the surrogate matrix spike compounds specified by the CLP SOW and Methods 8240 and 8270.

These surrogate compounds are measured from the laboratory preparation blank (reagent water) that is specific to each lot of sample preparations. Acceptance Limits for RMAL's LCS and SCS results are the same as the CLP specifications for matrix spike recoveries, matrix spike duplicate results, and surrogate recoveries. Please note that LCS's are prepared in reagent water while CLP matrix spike advisory limits are established for actual samples matrices.

Solid/soil/sludge samples will be prepared using the methanol extraction procedure for VOC's and the CLP SOW procedures for semi-volatile compounds. This should be acceptable for the proposed Appendix IX monitoring. RMAL uses continuous liquid-liquid extraction for the preparation of water samples, or groundwater, instead of the commonly used sepratory funnel liquid-liquid extraction for Method 8270. Review of LCS recoveries for the eleven semi-volatiles in reagent water indicates excellent recoveries for these 11 test semi-volatile compounds at RMAL. These recoveries at RMAL are better than the average recoveries demonstrated throughout the United States. RMAL does not appear to have calculated or updated QC warning and acceptance limits for their LCS and SCS QC audits.

RMAL is using CLP SOW criteria for acceptance, that are based on actual samples and weighted towards the sepratory funnel extractions; however, note that RMAL should obtain excellent extraction recoveries for waters.

III. A. <u>OBSERVATIONS and RECOMMENDATIONS for RMAL METHODS 8240 and 8270</u>

1. RMAL has not completed documentation of SOP's that was initiated in 1987 as required by their corporate QA Plan. SOP's for these 2 methods were 50% completed at time of the laboratory evaluation. The laboratory is following procedures specified by the CLP SOW 7/87 for organics and by SW-846, 3rd edition, with raw data collected consistent with these test procedure requirements, and has authentic instrument calibration compounds for the "extra" Appendix IX compounds. We should accept RMAL as being acceptable for the determination of Appendix IX contaminants listed in their February 1988 document (attached to the report) for the above 2 methods.

2. The QA Plan for the Modern Plating site, specifies Foth and Van Dykes laboratory personnel will be evaluating and assessing data, for project acceptability, from RMAL. RMAL routine data packages and reports do not contain analysis information sufficient for thorough data assessment and for a evidential file.

Recommendation: Foth and Van Dyke needs to implement procedures with RMAL to obtain raw data records of analysis, including records of instrument calibrations and hard copies of mass spectra, as part of RMAL deliverables. This was acceptable to RMAL personnel at time of the lab evaluation.

3. RMAL, as part of a corporate policy, subtracts laboratory blank results from sample results for each analysis run. See ENSECO Inc. QA Program Plan, June 1988. This is a corporate policy and is not necessarily the RMAL technical staff's desire. This practice is not acceptable. Region V, U.S. EPA allows a certain amount of common organic lab contaminants in a lab blank (methylane chloride, ketones, phthalate esters, etc.) but does not allow target compounds in lab and field blanks.

Recommendation: For the Modern Plating study, RMAL should provide lab blank results for each set of corresponding sample results as part of the raw data and QC Lot. Foth and Van Dyke should make the decisions whether any sample results are affected, or need to be deleted based on lab blank or field blank results.

RMAL should not report any data with lab blank results (for blanks carried through all sample preparation steps) being unacceptable, except for commonly accepted lab blanks (methylene chloride, phthalate esters, etc.). None of the suspected contaminates at the Modern Plating site are common lab contaminants. This EPA recommended practice should be worked-out between Foth and Van Dyke and RMAL as part of the raw data package.

4. We note that the ENSECO QA Program Plan, June 1988 specifies that soil sample results will be reported, as received, on a wet weight basis.

Recommendation: For the Modern Plating site, RMAL must perform a % solids determination on all samples received so as to convert "wet" sample results to a "dry" weight basis (103-105 ^{o}C). Foth and Van Dyke and RMAL should decide who is to perform any data reduction of mg/kg (wet weight) to mg/kg (103-105 ^{o}C). Region V EPA traditionally uses soil results reported on a dry weight basis.

5. The ENSECO QA Program Plan does not provide for matrix specific QA audits, or project specific QC audits. With each set of 20 or fewer samples, prepared for analysis by Method 8240 or 8270, Lab Control Standards are prepared and tested. Matrix spikes are not routinely tested, unless requested by a client as project specific QC.

   Note that the LCS's are prepared in reagent water and do not reflect sample matrix interferences and note that RMAL has yet to establish statistical limits for their LCS results (CLP SOW limits are used). The Modern Plating QA Plan endorses the use of matrix spikes in accordance with SW-846, 3rd edition.

Recommendation: Foth and Van Dyke will have to work out a matrix spike/matrix spike duplicate procedure with RMAL for each set of sample types submitted to RMAL. The matrix spike compounds of the CLP SOW are a generally acceptable set of matrix spike compounds since they contain a cross-section of the target compounds to be determined, and methodology capabilities are known from the CLP SOW. For any Phase II type monitoring, Foth and Van Dyke should certainly refine any list of matrix spike compounds and should certainly consider any organic contaminants known suspected at this time for Modern Plating site soils and sludges.

6. RMAL mass spectra tuning compound (BFB for Method 8240 and DFTPP for Method 8270) and a calibration standard (with SPCC and CCC criteria) is measured once per day per GC/MS instrument. SW-846, 3rd edition, specifies these checks are to be made every 12 hours as is done by the CLP SOW. RMAL indicated they would do these QC checks at the request of their clients. The Modern Plating QA Plan specifies use of SW-846, 3rd edition.

Recommendation: Foth and Van Dyke should see that these 2 audits are done by RMAL at least every 12 hours for each GC/MS instrument used and that acceptable audit results are reported with raw data package.

IV. METALS-ICP EMISSION SPECTROSCOPY and GRAPHITE FURNACE ATOMI ASORPTION

The ICP emission spectroscopy and graphite furnace test procedures and QC procedures used by the Contract Laboratory Program (CLP) Inorganics Statement of Work (SOW) are in place at RMAL. For the Appendix IX metals, RMAL determines 1) arsenic, lead, selenium, and thallium by graphite furnace atomic absorption, 2) mercury by cold vapor atomic absorption, and 3) the remaining 12 metals by ICP emission spectroscopy (Method 6010). Lead will be measured by ICP emission spectroscopy in soils if present in large enough concentration.

RMAL has a Jarrel Ash Model 61 ICP emission spectrometer (state-of-the-art) and has several graphite furnace instruments equipped with Zeeman background correction in the metals analysis group that determines Appendix IX contaminants. The instrumentation is quite acceptable and is capable of providing detection limits considerably smaller than the Practical Quantitation Limits (PQL's) of the July 9, 1987 Appendix IX List.

The ICP direct reading instrument is set-up to measure all elements required by the CLP SOW and as well as other elements. The staff is familiar and knowledgeable with proper interelemental correction factors and can make use of the powerful software available with their Jarrel Ash system. The laboratory determines detection limits and ICP linearity checks on a quarterly basis, as is required by the CLP SOW.

The reporting limits (of reported detection limits) established by ENSECO are generally equivalent to or smaller than the PQL's of July 9, 1987. The ICP system in use can report detection limits considerably less than the PQL's or ENSECO reporting limits; however, the ENSECO reporting limits are still acceptable, except in one instance. An ENSECO reporting limit of 0.2 mg/l may be used for nickel. The July 9, 1987 PQL is listed as 0.05 mg/l. An instrument detection limit of 0.02 mg/l can be expected. RMAL needs to justify a reporting limit of 0.2 mg/l for nickel and why it should not be smaller. Reporting limits used by RMAL for the 4 graphite furnace metals and mercury are 10 times smaller than the Appendix IX PQL's.

The QC program in place at RMAL uses digestion blanks, duplicate Lab Control Standards (LCS's), matrix spikes, and duplicate sample analysis with each set of 20 samples digested prior to analysis. ICP and graphite furnace analyses use proper calibration verification solution checks and continuing calibration checks every 10 samples. Method of standard addition checks are in place for graphite furnace measurements. RMAL uses control limits of 80-120% recoveries for the LCS's instead of the stated 75-125% recoveries of their present QA reports.

The RMAL metals analysis procedures are consistent with the requirements of SW-846 except in one instance. RMAL measures the ICP interference check sample (prepared as required by the CLP SOW) once per week, because of limited quantities of solution, instead of twice per 8 hours as required by Section 8.6.3 of SW-846 Method 6010.

RMAL can provide Foth and Van Dyke raw data for metals analyses including sample digestion logs and instrument read-outs in order to provide data assessment and evidential records.

IV. A. OBSERVATIONS and RECOMMENDATIONS for RMAL METALS ANALYSIS

1. RMAL has no documented SOP's for metals analysis. This is a weakness of the ENSECO QA program in that SOP's are required. The laboratory's analysis procedures and raw data records are consistent with the requirements of SW-846 and are acceptable for the determination of Appendix IX metals pending resolution of the Observations and Recommendations listed below.

2. Recommendation: As discussed for Methods 8240, and 8270, Foth and Van Dyke 1) will need to obtain raw data records in addition to the routine ENSECO analysis reports, 2) will need to obtain metals data that is not digestion blank corrected, 3) will either need to obtain soils results reported on a dry weight basis, or the % solids values necessary for this conversion, and will need to verify with RMAL any project specific QC audits necessary for each set of processed samples.

3. Recommendation: RMAL needs to justify why any reporting limit of 0.2 mg/l for nickel should not be smaller in concentration for waters. The detection limit is not critical for soil analysis for nickel. Nickel is an important contaminant at the Modern Plating site for groundwater.

4. Recommendation: Each set of samples measured by ICP emission spectroscopy should contain an acceptable measurement of an interference check sample.

5. Recommendation: Any unacceptably large digestion blank value for metals should cause the sample analysis results should not be corrected for these blank values, and the samples redigested for reanalysis as appropriate.

V. VOLATILE ORGANIC COMPOUNDS (VOC's) BY METHOD 601 and METHOD 602

Rocky Mountain Analytical Laboratory (RMAL), ENSECO provides for VOC analysis by purge and trap, gas chromatography analysis using EPA Methods 601 and 602 from the wastewater regulations defined by 40CFR136. These were proposed by Foth and Van Dyke and by RMAL to measure the VOC Method 601 List and VOC Method 602 list in Tables 2-3 and 2-4 of the Modern Plating QA Plan. Approximately 75 groundwaters are proposed to be submitted to RMAL by Foth and Van Dyke for the Modern Plating RFI.

Attachment A to this QAPP is the list of VOC's routinely reported by RMAL for Method 601. Note that 2 Freon VOC's would be reported by RMAL, that are not part of QA Plan Table 2-3.

Attachment B to this QAPP is the list of VOC's routinely reported by RMAL for Method 602. Note that the dichlorobenzenes of Table 2-3 are reported by Method 602 and not by 601. Note that the 3 xylene VOC's are reported by RMAL Method 602 in addition to the aromatics of QA Plan Table 2-4.

The dichlorobenzenes are sensitive to the detectors used for both Method 601 or Method 602. The xylenes would be important to the Modern Plating site if aromatic hydrocarbon contamination is present.

RMAL has implemented Methods 601 and 602 through use of a single analytical system instead of using 2 different purge and trap, GC systems as Methods 601 and 602 were originally written. The effluent from the packed, gas chromatography column (1% SP-1000 on Carbopack B) of Method 601 leds to the Photoionization Detector (PID) of Method 602 and then to the Hall Conductivity Detector of Method 601. The PID is a non-destructive detector sensitive to the unsaturation in halogenated ethenes, halogenated propenes, hydrocarbon aromatics, and halogenated aromatics of Methods 601 and 602. The Hall detector is sensitive and specific to chlormated, brominated, and iodinated compounds of Method 601. The Hall detector is a destructive detector.

Purge and Trap GC instrument read-outs are obtained for both Method 601 and Method 602 by using a single purge and trap injection and a common GC column, but 2 different method specific detectors. This is a modern practice common to many laboratories, and is now a recognized VOC methodology (EPA Method 502.2) for drinking waters.

RMAL has 4 purge and trap GC systems set-up for their version of Method 601/602. The laboratory also has 2 similar systems, each with a RESTEK capillary column, instead of the 1% SP-1000 liquid phase packed column, for drinking water analysis (EPA Method 502.2) for resolving and determining 60 regulated VOC's in drinking water. This Method 502.2 system has the capability to be used for confirmatory analysis and identification of VOC's presumptively identified by Method 601/602. Instrumentation and equipment at RMAL are excellent for Method 601/602.

RMAL calibrates their Method 601/602 initially with a 5 point calibration curve made from 0.00, 0.50, 1.0, 5.0, 20, and 60 ug/l concentration VOC standards. Each standard solution, fabricated from Supelco stock standards contains all the VOC's to be reported by RMAL for Method 601/602. The Nelson data systems used for each GC systems compensate for any non-linearity of detector response for each VOC on a specific detector. All VOC's to be reported by Method 601 are calibrated on the Hall detector. All VOC's to be reported by Method 602 are calibrated on the PID.

This system is acceptable in concept, except in one instance. Attachment C to this report provides a retention time behavior for a large number of VOC's on the SP-1000 column used for Method 601/602. It is from the Minnesota Dept. of Health Method 465-B. Attachment D to this report provides the same retention time behavior for the large number of regulated VOC's that are now part of the 40CFR141 EPA drinking water regulations. Attachment D is from EPA Method 502.1.

For RMAL Method 601, 4 pairs of VOC's are known to co-elute on the SP-1000 column. The Hall detector response can not resolve or differentiate between the following triplits or pairs of VOC's in Attachment A

1. dichlorodifluoronmethane and vinyl chloride
2. cis- and trans- 1, 2-dichloroethene
3. dibromochloronethane, trans- 1, 3-dichloropropene, and 1, 1, 2-trichloroethane
4. tetrachloroethane and tetrachloroethene

The data reports from RMAL for Method 601 will state none of the co-eluting VOC's are present if none are, detected. If any of the co-eluting VOC's are detected the RMAL data reports will state these VOC's could be 1 of 2 or 3 possible VOC's with no confirmation of identity of any of them unless a client pays for this confirmation by Method 502.2.

It is most proper to report the cis- and trans- isomers of 1, 2-dichloroethene as their sum unless a client wishes their true identity. The other 3 sets of VOC's require confirmaton for individual identity by the Hall detector.

Another problem is that the different co-eluting groups provide different Hall detector response for each individual VOC. If quantitation results are reported for any co-elution group, they are estimated values since each co-elution group instrument response is a weighted average of each VOC. Initial calibration by a 20 ug/l standard will provide a Hall detector response that is the sum of 20 ug/l vinyl chloride and 20 ug/l of the Freon co-elution VOC; therefore, calibration is based on the weighted response of vinyl chloride and the Freon VOC. Three of the above 4 groups have this quantitation problem. Cis-and trans- 1, 2-dichloroethene should have the same Hall detector response per unit weight for each isomer.

Other laboratories, which use Method 601/602, can calibrate the system for co-eluting VOC's by using 2 or 3 sets of calibration standards, so that the original 5 point calibration curve is composed of 10 or 15 standard injections, instead of the 5 used by RMAL. RMAL is taking the most cost-effective (from their stand point) way of instrument calibration for co-eluting VOC's; however, EPA Method 601/602 requires confirmation of identity of VOC's if the samples are of unknown composition. Note that both EPA Methods 601 and 602 provide conditions for second column confirmation, or suggest GC/MS for VOC confirmation.

Attachments C and D to this report provide retention time behavior for a larger number of VOC's than measured by Methods 601 and 602. Note than when allyl chloride (3 chloropropene), and any of the possible dichloropropanes and possible dichloropropenes are present, the number of co-eluting VOC's increase over what is exhibited by RMAL's version of Method 601. Note that a co-elution problem exists fo o-xylene and p-xylene by RMAL Method 602. This should not be a problem in that the sum of the isomers are reported. This xylene co-elution problem is common to many VOC methods; however, this co-elation does not have the significance of not distinguishing between a Freon and vinyl chloride.

For the Modern Plating site, tetrachloroethane and vinyl chloride are known site specific contaminants. 1,1,2-tricloroethane could also be a possible contaminant. The dichloropropanes and dichloropropenes are not a strong possibility. The Modern Plating RFI does not lend itself to RMAL's version of Method 601/602 for tetrachlorethane and vinyl chloloride. RMAL indicated if Foth and Van DYke wanted to pay for confirmation identity by Method 502.2, confirmation could be provided.

After initial instrument calibration RMAL provides systematic operational and QC practices to maintain accuracy and precision of measurement of the analytical GC systems by the following manner:

1. A 30 ug/l bromochloromethene internal standard is used for the Hall detector. A 30 ug/l trifluorotoluene internal standard is used for the PID. All calibration standards and sample quantitaions are based on this internal standard response.

2. Each day of use, a 20 ug/l calibration standard is measured to verify the initial standard curve is still valid.

3. A Lab Control Standard (LCS) containing 5 VOC's for the Hall detector and 5 VOC's for the PID is tested. This is an independently prepared standard to assess accuracy and precision of measurement at the 5 ug/l concentration level. These results are reported with each QC lot of sample results. One VOC (chlorobenzene) is common to each of the above LCS solutions, so that Hall and PID results can be compared.

4. A Surrogate Control Standard (SCS) containing 20 ug/l of each internal standard VOC (2 in number) is measured to check recovery. The internal standard responses are monitored throughout the sample analyses.

5. RMAL participates in EPA Water Supply Performance Samples and provides generally acceptable results, except where co-elution problems exist.

6. Acceptance limits for the above QC Audits were reviewed but found not acceptable, as discussed later in this report.

7. RMAL provides reporting detection limits of 1 or 2 ug/l for each of the VOC's. Documented SOP's for Method 601 and Method 602 at RMAL provide estimated detection limits, based on experimental data of 0.5 ug/l or less, with some of the late eluting VOC's being 1.0 ug/l for Method 601. RMAL can provide detection limits less than the reporting limits common to ENSECO, Inc.

V. A. OBSERVATION and RECOMMENDATIONS

1. RMAL has the capability to provide acceptable VOC data by their Method 601/602 methodology, if it or Foth and Van Dyke desire to do so.

2. RMAL has the capability to provide confirmatory identity of detected VOC's using Method 502.2 in addition to Method 601/602. RMAL will provide confirmatory analyses if a client pays for it.

3. U.S. EPA Region V's policy is to state that RMAL's present 601/602 methodology is not acceptable unless confirmation is done, as required by EPA's reference methods. Known site-specific VOC's at the Modern Plating site (tetrachloroethane and vinyl chloride) mandate confirmation be done and quantitation be based on standards that are not weighted averages of 2 or more standards. Other VOC co-elution problems may exist for the site, but these will not be known until future analytical work is done.

4. RMAL's SOP's are 95% complete and accurate and generally reflect current situations for Method 601/602.

5. Review of several sets of VOC data at RMAL indicates generally accurate QC audit results are obtained. VOC results of less than 20% error should be obtained for results exceeding 10 ug/l. Errors of less than 20 to 40% should be obtained for VOC concentrations less than 10 ug/l. These accuracy limits approximate errors allowed during U.S. EPA interlaboratory testing of unknown reference samples for VOC's. RMAL's QA program does suffer because arbitrary acceptance limits have been established by ENSECO is described below for Method 601/602 QC audits:

a. LCS - 60-140% Recovery for Hall detector
77-123% Recovery for PID

b. SCS - 20-160% Recovery on each Detector for Internal Standard

The QC audit results reviewed were much better quality than these limits. Allowing a 60% recovery does not seem reasonable for the LCS, to maintain accuracy at allowable errors of 20% or less. We can not accept 20% or 60% recoveries for the internal standard (which is reported as a surrogate control). The 601/602 methodology does not lend itself to use a SCS as an accuracy check, then use the surrogate as an internal standard for quantitation purposes. Lab personnel were reviewing internal standard responses in each sample after initial instrument calibration, but statistical estimates had not been formalized.

Recommendation: The RMAL QA staff should statistically evaluate recent LCS and calibration check solution (20 ug/l of each VOC) results and see that new acceptance criteria could not be estimated. The SCS audit should possibly be deleted.

An audit for internal standard response for each sample should be established and formalized with limits in the range of 80-120% or 75 to 125% being established. A calibration check standard needs to be tested at the end of each set of daily analyses. Current QC audits do not reflect reality because they appeared worse in quality than the more accurate data actually being obtained, and current QC audit acceptance limits do not reflect program needs for accuracy. Improvements and updates in QA program for Method 601 and 602 are needed.

6. Summary - Before Method 601/602 is used by RMAL, 1. confirmation of VOC identity and; quantitation of VOC results against proper, non-mixed standards must be established, and 2. its QA program be updated to provide meaningful QC audit acceptance criteria.

c.c. D. Payne, CRL
J. Adams, MQAB
Attn: M. Long

Call back ~~20~~ MINS

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5

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FROM: David A. Payne, Chemist

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PAGE 1 OF ~~13~~ 14 PAGES

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION V

DATE: APR 26 1990

SUBJECT: Laboratory Evaluation - ENSECO/Rocky Mountain Analytical Laboratory (RMAL), Denver CO for Johnson Wax, WI
RCRA Facility Investigation (RFI)

FROM: Curtis Ross, Director Chuck Elly for
Central Regional Laboratory

TO: William E. Muno, Chief
RCRA Enforcement Branch

ATTN: Glenn Sternard

Attached is the final lab evaluation report for ENSECO/RMAL, Denver, CO. The points discussed are:

1. A restatement or summary of changes in compounds to report for RMAL analyses.

2. Problems (possible) in adding 9 phosphorus pesticides to Method 8270, with a request for justification (promised by RMAL).

3. Further details (as requested by CH2M-Hill in their letter of March 2, 1990) on PQL's used for four compounds of Method 8270, and for water miscible volatiles of Method 8240. New PQL's proposed by RMAL for the 9 phosphorus pesticides appear too large for Method 8270. Recommendations are made for possible changes.

4. Operations of GC Methods 8140, 8150 and 8080 with recommendations for reporting limits. The DDT and endrin degradation checks are discussed, as well as standard preparation(s) for Method 8150, and calibration curves for all of these procedures.

5. Sample extract clean-ups and surrogate and matrix spike compounds. These should be principal topics for any technical meeting. We should find as reasonable an approach as possible.

6. Adequacy/Inadequacy of SOP's for ENSECO/RMAL were discussed in previous February 15, 1990 correspondence from CRL to RMAL. CH2M-Hill in letter of March 2, 1990 said that these deficiencies will be addressed.

7. The question of benzene methodology has been referred to QA Section of Region V, and the CRL and QA Section agree with the recommendations of ENSECO/RMAL and CH2M-Hill.

With the memo reports of April 4 and 20, 1990 to you (concerning non-Appendix IX issues), and with resolution of the above points, we believe ENSECO/RMAL will be appropriate and acceptable for the Johnson Wax RFI. We do not yet have an acceptable alternative to the current extraction solvent system for Method 8150 analysis of soils.

cc: G. Schupp, QA Section-EPA
D. Payne, CRL-EPA
E. Evangelista, CRL-EPA
K. Gunter, CRL-EPA

I. INTRODUCTION

During January and February 1990, the Region V Central Regional Laboratory (CRL) reviewed the analysis records (Appendix IX), for 1 water (field blank), 2 soils, and 2 wastes as provided by ENSECO/RMAL. These were reviewed for conformance with ENSECO's QA Plan SOP's and QC practices, for conformance with SW-846 requirements, and for technical merit. ENSECO/RMAL was able to provide indexes to the raw data records for calibration curves, daily continuing standards, certain blanks, and samples or sample extracts. Questions concerning the data records were FAXED to RMAL on January 16, January 26, January 30, and February 8, 1990. An initial evaluation of ENSECO's Appendix IX procedures was FAXED to RMAL and to CH2M-Hill on February 15, 1990 subsequent to telephone conference calls between the CRL and RMAL. ENSECO/RMAL should be complimented for their cooperation in 1990. RMAL and CH2M-Hill, in a letter dated March 2, 1990 have voiced either their concerns or agreements with items in the February 15th evaluation. This memo report is now a finalized report for ENSECO-RMAL's analysis procedures for appendix IX and associated analyses for the Johnson Wax RFI. This memo report does not address dioxin/dibenzofuran analyses by method 8280 to be done by ENSECO/CAL.

II. APPENDIX IX COMPOUNDS OR PARAMETERS

A. The following compounds should be noted as not being detectable by ENSECO/RMAL using GC/MS methods.

1. 4-Nitroquinoline-1-oxide
2. p-Phenylenediamine
3. Hexachlorophene
4. Pyridine (soils)
5. 1,4 Dioxane at a D.L. of 100 ug/l in water. 1,000 ug/l standards of 1,4 Dioxane did not provide sufficient sensitivity to warrant using a D.L. of 100 ug/l.

6. Ethyl Cyanide at a detection limit of 5.0 ug/l. The first 4 compounds are a fact of life. Many labs can not detect these compound. There is disagreement between the CRL and a RMAL on D.L. for 1,4-Dioxane. The ethyl cyanide was not discussed previously with RMAL.

7. Famphur and Dimethoate.

B. The following compounds should be added to data reports for RFI.

1. Cis-1,2-Dichloroethene (Presently part of RMAL Method-8240 calibration standard)

2. α&γ-chlordane - This can be added to Method 8080 calibration standards based on QA Plan objectives for the site.

C. Fluoride can be deleted from RMAL's list of Appendix IX compounds since it is not part of Appendix IX.

D. Method 8140 Pesticides (Phosphorus Containing)-RMAL-plans to add these 9 phosphorus containing compounds to the list of analyses to be determined by Method 8270. The 9 compounds are currently part of the continuing calibration standard solution used for instrument calibration in Method 8270. Method 8270 has been deemed acceptable and more cost-effective than the more sensitive Method 8140. One method (Method 8140) will then be eliminated for Appendix IX determinations.

Two of the 9 compounds are believed to be non-detectable by method 8270:

1. Dimethorate - The high pH of the initial BNA extraction can destroy this compound, based on U.S EPA method validation studies. See June 5, 1987, report copy provided to ENSECO/RMAL

2. Famphur - GC/MS response appears to be as bad as hexachlorophene; however, its GC response for Method 8140 is not very good based on data package review.

Since writing the February 15, 1990, initial lab evaluation report, the CRL has spoken with different Appendix IX laboratories. Certain lab personnel are concerned about the high pH values in the BNA water extraction for the phosphorus pesticides of Method 8140. The June 5, 1987, method validation of EPA for the extraction of the 9 compounds used a seperatory funnel extraction for the waters. RMAL is using a continuous liquid-liquid extraction of 16 hours at high pH. RMAL has indicated, during telephone conference calls, that MDL studies have been done by Method 8270 for the 9 compounds.

1. Recommendation:

RMAL should provide recovery data for BNA extraction of the 9 compounds using the continuous liquid extractor. This is a new comment from Region V, but it is reasonable. The initial extraction should be done at a high pH value (if this has never been done). RMAL should justify recovery of these compounds by Method 8270 while Method 8140 has only a neutral pH extraction of waters. In a telephone conversation with RMAL on 4-3-90, RMAL personnel promised to provide these recovery data for the 9 pesticides of Method 8140.

2. Recommendation:

Our February 15, 1990 memo made no recommendations for matrix spike compounds additional to the CLP program. We now recommend the additon of 2 or 3 of the Method 8140 compounds to the present list of compounds used as matrix spikes for Method 8270.

So far we definitely question the validity of Method 8270 for 2 of the 9 Method 8140 compounds. One of these 2 compounds is unimportant (Famphur). Both should be noted as undetectable in data reports unless dimethoate recoveries can be demonstrated acceptable. RMAL should provide data to justify recoveries of the remaining 7 compounds, using the liq-liq extraction, if and when available.

E. N-nitrosodiphenylamine and diphenylamine can not be differentiated (in the same way as m-cresol and p-cresol can not be differentiated) by Method 8270. These compounds should be noted as "either/or" in final data reports.

III. GC/MS INSTRUMENT OPERATION - METHODS 8240 AND 8270

The data package reviewed provided examples of excellent GC/MS operations. Procedures in effort follow and are consistent with the technical requirements of the CLP Statement of work. ENSECO-RMAL was the first lab reviewed for RCRA programs where no errors have been observed for these operations. Appendix IX compounds have been properly apportioned or selected for the 2 methods, however, each Region V RFI will need to determine whether 8140 should be used for the 9 pesticides, or whether Method 8270 will be appropriate. This is a site-by-site decision. The decision has been made to use Method 8270 for the 9 pesticides.

Calibration of the 2 Methods and instruments are done using a 5 Point Calibration Curve for reference for the CLP SOW TCL compounds. Each day after initial MS tuning and blanks, two standards are used to update the instrument response factors and retention times using authentic standards. One standard contains the CLP TCL compounds and one the extra Appendix IX compounds (a 5 Point calibration Curve is not available for the extra Appendix IX compounds, but this has been acceptable to Region V for any "extra" Appendix IX compounds that ar not site-specific contaminants).

Surrogate recoveries were available for Method 8240 because they were added after extraction of the wastes with methanol. Both surrogates showed little, if any, deviation from 100% recovery. Internal standards response was relatively constant for both Methods 8240 and 8270 and of excellent quality. The in-house Lab control Standards and Surrogate control Standards for both soils and waters did not significantly differ from 100% recoveries.

These are indicative of excellent instrument operation and calibration for Appendix IX compounds.

The one difference in instrument operation between EPA programs and ENSECO's industrial clients is that tuning checks and continuing calibration standards are not determined every 12 hours for industrial clients as they are one for EPA programs.

On page 2 of CH2M-Hill's letter of March 2, 1990 our questions on the preparation of standards for Methods 8240 and 8270 have been resolved, by informal discussions with RMAL staff concerning the proprietary nature of the daily calibration standard used for the extra Appendix IX compounds.

On page 2 of the March 2nd letter we will now detail our concerns with the consistency of Reporting Limits or "PQL's". Most PQL's are consistent or the same as CLP SOW CRDL requirements. For well-behaving compounds (chromatography and MS response) the SOW requirements are exceeded by RMAL because of their excellent instrument operations. We will list below certain observations on Appendix IX compounds.

A. Method 8240

Compound	Daily Std. Conc. (ug/l)	Reported PQL (ug/l)	Area Counts for Quant Ion (SI515 or 517) for daily std.	Response Factor
Acetone	50	25	984	0.05
Acetonitrile	100	100	2,400	0.119
Acrolein	50	100	1,000	0.05
Acrylonitrile	50	100	3,100	0.136
Isobutanol	100	100	2,000	0.03
2 Butanone	50	25	1,600	0.02
Ethyl Cyanide	50	5	1,700	0.110
MIBK	50	10	7,000	0.12
2 Hexanone	50	10	5,600	0.10
1,4 Dioxane	1000	100	3,300	0.011

B. Method 8270

Compound	Daily Std. Conc. (ug/ml) or (ug/l)	Reported PQL (ug/l)	Area Counts for Quant Ion (STDR 508 or 571)	Response Factor (STDR 508) (STDR 571) (STDR 773) (STDM 812)
Aramite	50	10	3,800(#1)	0.05.-0.08
Isoafrole	50	20	3,300*(#1)	0.06
Petachloronitro-	50	50	5,7000.	0.075

benzene				
Sym-Trinito-benzene	50	10	5,300	0.06
Demethoate	50 **	Not Det.*	21,000	0.31-0.39
Disulfton	50**	50*	43,000	0.52-0.62
Parathion	50**	50*	14,000	0.17-0.21
Famphur	50**	100*	No Response	0.0
Methyl Parathion	50**	50*	17,000	0.24-0.28
Phonate (Thimet)	50**	100*	15,000	0.18-0.20
Sulfotepp	50**	50*	15,000	0.12-0.16
Thionazin	50**	50*	14,000	0.26-0.31
0,0,0-etc.	50**	50*	1,000	0.13-0.16

* - Data Faxed to CRL on 2-14-90
** - 1989 Data may not be applicable to 1990.

Review of the 1989 data package showed RMAL to report the presence and concentration of all MS "hits" even if the concentration was less than the "PQL". Data reduction would then change these values to "less that PQL" even though acceptable mass spectra are present (The CLP will report these low concentrations with a "J" qualifier).

The above data for Method 8240 water soluble-low purge efficiency volatiles show no consistency between area counts of mass spectra quant ion, PQL used, and response factors. Area counts for good acting compounds with response factors greater than 0.30 or 1.0 should certainly be detected below their PQL of 5 or 10 ug/l. Also, why does acetone have a PQL of 25 ug/l while acrolein is reported using a PQL of 100 ug/l and 1,4 Dioxane is reported with a PQL of 100 ug/l?

For Method 8270, is it reasonable to expect a PQL of 10 ug/l is appropriate for sym. trinitrobenzene? Why are the PQL's set at 50 or 100 ug/l for the 9 Method 8140 pesticides when their response factors are significantly larger than 0.10 or 0.20? By changing from Method 8140, PQL's are increased 100 fold through use of Method 8270.

Recommendations:

1. Report all positive MS "hits" with acceptable mans spectra as recommended by CH2M-Hill in their letter of March 2, 1990.

2. Justify the large PQL 's for 7 of the 9 Method 8140 pesticides. Whey don't they approximate 10 ug/l?

3. Significantly increase the PQL for 1,4 dioxane and Ethyl Cyanide.

4. Justify why Daily Std concentrations are lower than their PQL's for Acrolein, and Acrylonitrile. How can you calibrate or

update an instrument using concentrations smaller than their reporting limits?

5. Is there a rationale for selection of PQL's for the water soluble compounds of Method 8240?

6. Why is the PQL set at 10 ug/l for sym. trinitrobenzene?

7. Justify whey Famphur is reported at all. Does RMAL have extraction recovery data for Dimethoate to justify its non-detection other than the EPA validation report?

IV. GC INSTRUMENT OPERATIONS - METHODS 8080, 8140 AND 8150

The instrumental operations for ENSECO Methods 8080, 8140, and 8140 are fairly straight forward, after review of a "typical data set". Five point calibrations are provided for the CLP and TCL pesticides of Method 8080. Single standard solutions are injected for chlorobenzilate, isodrin, kepone, and the multicomponent Arcolors, tech. chlordane, and toxaphene. External standards are used to quantify these compounds in sample extracts. ENSECO is to be complimented on their thorough documentation of Rention Time windows and their use in their Nelson Data System for the CLP TCL pesticides.

1. Recommendation:

   ENSECO needs, as a reference, 5 point calibration curves for the determination of linearity, acceptable instrument response, detection limits, etc., for the 3 Appendix IX pesticides, α-chlordane, α-chlordane, and at least 4 of the routinely determined Acrcolors (1242, 1248, 1254, and 1260). We would encourage use of the Nelson Data System for these latter compounds, but do not require it. It is helpful to use the data system to plot the reference calibration curves, instead of reviewing individual calibration factors.

2. Recommendation:

   ENSECO should consider that either DDT or endrin degradation must be <20% when pesticides are reported. We would encourage these degradations to be smaller. We realize that automated instrument operation does not allow these determination to be made prior to sample analysis; however, SW-846 Method 8080 mandates the degradation be less than 20% for either compound.

The instrument operations for Method 8140 were routine; however, 3 of the 9 compounds were inappropriate concentrations for their 5 point calibration curves. This is documented in case the methodology is used for other projects.

ENSECO's Method 8150 had a 5 point calibration curve, of excellent

quality. The SOP specifies the standards are prepared in-house using the methylation reagent that is used for samples. Telephone conferences with ENSECO indicated the standards (for the 3 herbicides of interest) are purchased methyl esters.

3. Recommendation

SW-846, 3rd edition, and ENSECO's in-house SOP both specify preparation of standards using the in-house methylation reagent - diazomethane. This needs to be resolved. If standards are to be purchased as methyl esters, controls will need to be carried through each set of sample preparations.

4. Observation:

In the "typical" data set for Method 8150, a typical 5 point calibration curve was constructed, using methyl esters. The SOP for Method 8150, specifies a 4 point calibration curve of 3 standards with the origin being the 4th point. No calibration curve was submitted for our evaluation. The Nelson Data System for dual acquisition from 2 columns supplied with the data package gave information as to the consistency of the response factor (RF) through the entire calibration range, except for Dinoseb on channel 1 and DCAA and Dinoseb on Channel 2. RF's for all other compounds were consistent. A calibration curve or consistent RF would be helpful in estimating Reporting Limits for the Herbicides, along with herbicides prepared from the in-house methylation reagent.

5. Statement:

ENSECO is to be complimented for use of their dual (simultaneous) capillary columns for simultaneous confirmation, identification, and quantitation.

V. <u>REPORTING LIMITS FOR METHODS 8080, 8140, AND 8150</u>

A variety of reporting limits or "PQL's" were questioned or discussed in previous correspondence for the GC methods. The PQL's appear arbitrary for ENSECO.

1. <u>Recommendation:</u>

The final reporting limit used should be based on the amount reasonably detected in the sample extract (ug/ml), multiplied by the extract volume (10ml for Method 8080 or 1ml) for Method 8270), multiplied by any extract dilution, and related to 1 liter of water or 10 to 30g of soil aliquot. If no interferences are present, the reporting limit should approximate or bear some relation to the lowest concentration value measured in a compound's 5 point calibration curve. During sample analysis for the RFI, reporting limits or sample

concentrations identified from 2 columns should be reported through the above calculations.

VI. SAMPLE EXTRACT CLEAN-UPS

For the "typical" (or "non-typical") data set reviewed, interferences in sample extracts of non-typical soils or wastes had a bigger effect on sensitivity of analysis than any other factor. Extracts were not cleaned for Methods 8270 or 8150. Interferences caused dilution of the extract (so as to dilute out surrogates) for Method 8080 even though the extracts were taken through the 50% ether fraction on Florisil. Interferences in sample extracts had a larger negative effect on data quality than any other factor. The excellent instrumentation of ENSECO could not be used to its full capability due to over-dilution of extracts to minimize interferences.

Decisions for surrogate concentrations, matrix spike concentrations, and whether to clean-up an extract appear the responsibility of the sample preparation groups at ENSECO. If clean-ups are not done, but extracts still need to be diluted, or cleaned up, the extracts were not recleaned for the "typical" data set.

1. Recommendation

   If sample extracts are "dirty" or exhibit significant interferences with no clean-up they should be retested after cleaning. This is a non-negotiable issue.

2. Recommendation:

   We would encourage ENSECO to use gel permeation clean-ups of soil extracts,as they do for the CLP, for Method 8270. We can not require it at this time since we do not know the effect of gel permeation on the extra Appendix IX compounds.

3. Recommendation:

   The use of 6% ether or 15% ether/hexane elutions of soil or water extracts can easily contain less interferances than the 50% ether/hexane fraction. The use of a 6% ether/hexane fraction will contain most of the chlorinated hydrocarbon pesticides and Arclors of interest for Method 8080. See page 4 of 14 from our memo report of February 15, 1990. While we can not argue with "acceptable" results obtained from a combined 6%, 15% and 50% ether fraction, we believe a 6% and 15% fraction is in order when the combined fractions show or exhibit gross interferences.

A 6% ether/hexane fraction is necessary if silicic acid chromatography is to be used to separate pesticides from PCB's

We would encourage ENSECO and CH2M-Hill to find the most reasonable clean-up for the naphtha contamination expected.

4. Recommendation:

We have provided the silicic acid separation of pesticides and PCB's to ENSECO-RMAL. It was intended for the surface water sediments, and not necessarily for all soils; however, it mandates the use of a 6% ether/hexane fraction from Florisil before it can be used. If the 6% fraction is used, the PCB content present will mandate whether the silicic acid is necessary. The question of whether to use silicic acid, is or not is moot, since we do not have agreement on the 6% ether fraction from Florisil.

5. Recommendation:

We assume and recommend that ENSECO will clean all soil extracts for Method 8150. Evaluations of other Region V RFI labs have found that Method 8150 extracts of waters and soils definitely need "clean-ups", or elimination of interferences.

6. Recommendation:

We hope that any technical meeting between ENSECO-RMAL, CH2M-Hill, and Region V will define reasonable extract clean-ups for the planned RFI. We are not yet in agreement on extract clean-ups.

7. Recommendations:

Any data package received by CH2M-Hill should have a prep-log for sample extracts, with clean-ups tied and documented to specific SOP's.

VI. MATRIX SPIKES AND SURROGATES

Discussions on evaluation of the data set from ENSECO-RMAL centered on appropriate surrogates and matrix spikes. Our previous comments were not specific but centered on over-dilution of matrix spikes and surrogates, in sample extracts for soils and wastes.

1. Recommendation:

Add 2 or 3 of the Method 8140 pesticides (phosphorus) to the matrix spike mixture for Method 8270.

2. Recommendation:

   Consider increasing the matrix spike and surrogate concentrations for Methods 8270, 8080, and 8150 if they are being diluted -out for soil extracts. Use medium conc. spike levels for medium concentration samples dilutions.

3. Recommendation:

   Use tetrachloroxylene and decachlorobiphenyl as surrogates (advisory) for Method 8080. The DBC surrogate is a waste of time to represent florisil clean-ups using the 6% and 15% ether fractions.

4. Recommendation:

   ENSECO-RMAL needs to finalize its use of either DCAA or 24 - DB as a surrogate for Method 8150.

5. Recommendation:

   The data resulting from "LCS, "DCS," and "SCS" in-house controls should be part of any data package sent to CH2M-Hill; however, any such reported data should be documented in such a way that the data user can determine whether it is a solid or water type, whether it was prepared in the same lot of samples analyzed, and whether it was tested on the same instrument and same day as the samples of interest.

   We consider these control samples to be of use, but that their acceptance criteria be based on in-house statistical estimates and not on CLP criteria developed from actual samples, taken through all extract clean-ups. These control samples should not replace site-specific surrogates and matrix spikes.

VIII. ADEQUACY OF SOP'S

Our memorandum report of February 15, 1990 documented the inadequacy of SOP's provided for the RFI. The organics were either out-of-date or provided several options, some of which were confusing. Inorganics SOP's did not exist for the RFI QA Plan. CH2M-Hill comments on page 4 of their March 2, 1990 letter addressed these issues adequately. SOP's will be rewritten, or written to reflect reality.

VIX. MISCELLANEOUS ITEMS

1. The response of March 2, 1990, last "bullet" on page 3, concerning benzene is an appropriate compromise. The intent of determining the 8 regulated SWDA volatiles by 601 or 601/602 was to determine benzene by the sensitive PID detector methodology of Method 602 or 502.2, or of the methodology with

the PID and Hall detectors in series. This issue has been discussed further with the QA Section, Region V. Use of Method 8240 with an IDL of 2.5 ug/l for benzene will be appropriate for Round 1 monitoring of groundwaters.

2. We understood the ICP interference check sample is being used. It was not in the data that were reviewed.

3. We noted a severe interference for tin during soil and waste analysis by ICP emission spectroscopy. We believe this could be due to an spectral interference from sulfur. We do not know if ENSECO can correct for it, or provide a different type of background correction for tin.

4. We have never received the interelemental correction procedures originally requested. It is too late to do anything about it at the moment one way or another. Also, tin is not the most important element for the RFI. The project is too far along for this to have any effect. We should be concerned more on the SOP's that will be documented for the ICP emission spectroscopy.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
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FACSIMILE REQUEST AND COVER SHEET

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MACHINE NO.

VERIFICATION NO.

TO: Joe Sandrin - CH2M-Hill

FROM: David A. Payne, Chemist

PHONE NO. (312) 886-1970 (Office)
(312) 886-2591 (FAX)

MAIL CODE 5SCRL

OFFICE LSSS, CRL, Region V, EPA

DATE 4-26-90

NUMBER OF PAGES, INCLUDE COVER SHEET 19

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ENVIRONMENTAL SCIENCES DIVISION

WILLIAM H. SANDERS III, DIRECTOR

PAGE 1 OF 19 PAGES

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION V

DATE: APR 20 1990

SUBJECT: Letter of March 2, 1990 from CH2M-Hill for Johnson Wax RFI Concerning Submittals Separate from Appendix IX

FROM: Curtis Roes, Director Chuck Elly for
Central Regional Laboratory

TO: William E. Muno, Chief
RCRA Enforcement Branch

ATTEN: Glenn Sternard

This memo transmits comments on CH2M-Hills submittals of
1) February 21, 1990 for Hazleton special pesticides
2) January 16, 1990 for ENSECO/RMAL Naphthas
3) January 19, 1990 for ENSECO/RMAL General Chemistry

Draft comments were previously provided to Mr. Sternard, to ENSECO/RMAL, and to Joe Sandrin, CH2M-Hill on April 1, 1990. Comments are also provided for ENSECO's letter of November 10, 1989 concerning silicic acid. We believe that silicic acid is appropriate when interfering PCB's are present. Chlorinated hydrocarbon pesticides can be determined but only if the 6% ether/hexane fraction is available. The need for silicic acid will be apparent if PCB's are present, but we can not use it if we have disagreement on the need for use of the 6%, 15%, and 50% ether florisil fractions for Method 8080.

The Central Regional Laboratory (CRL) Region V has reviewed the following submissions of Hazleton Laboratories, CH2M-Hill, and ENSECO/RMAL for the SC Johnson Wax RFI. Questions concerning these comments should be directed to David Payne at (312) 886-1970.

A. Letter of February 21, 1990, from CH2M-Hill documenting SOP's (2), for the determination of 3 specialized pesticides in water. No soils/solids are to be determined.

The SOP's are generally acceptable and are consistent with the results of the CRL lab evaluation of Hazelton in September 1989. Two items should be addressed in SOP and/or QA Plan prior to final approval.

1. The SOP's should each specify the working concentration range of the method through use of the five or more calibration standards (in ug/ml) used for the reference calibration curve of each method. These then can be related to the water concentrations via the final extract volume. A daily calibration standard (in ug/ml) should also be specified. The final MDL reported (in a "clean" sample matrix) should bear some relationship to the lowest standard of the calibration curve (used as a reference).

2. The following method detection limits (MDL's) were provided by Hazleton. Also listed are Reporting Limits of QA Plan (Table BA-8)

Compound	Hazleton MDL (ug/l)	CH2M-Hill Reporting Limit (ug/l)
Baygon	1.5	50
Dursban	0.2	2
Vapona	0.3	2

If sample results indicate a concentration at or near the MDL, they should be reported as actual sample results. The CH2M-Hill reporting limits must not be taken as the sample detection limits. Interferences in sample extracts may preclude use of MDL's. In this case, the calibration curve can be used to establish reporting limits. You may want to consider the Reporting Limits as objectives for real samples with interferences, but also note that matrix and surrogate spikes will be used that are the same concentration (or less) as the Reporting Limit. The matrix spike and surrogate concentrations may have to be increased in concentration.

B. Letter of January 16, 1990, from CH2M-Hill documenting approach of ENSECO/RMAL for Naphthas (Isopars K,L, & M) in both water and soil.

We find this approach basically acceptable. We do have certain questions below; however, we find the approach systematic, reasonable, and technically sound. Reasonable estimates of Naphtha concentrations and their identity(s) will be obtained. ENSECO/RMAL must be commended on this technology.

We do have the following questions or comments on the procedure.

1. The November 13, 1989 Technical Memorandum of ENSECO/RMAL is not an SOP, or a detailed procedure. We assume that either 1 liter of water is extracted 3 times with methylene chloride, or 30g of soil is sonicated 3 times with methylene chloride and the extract concentrated to 10ml. If highly contaminated, the extract is diluted so the FID response of the final extract volume fits the 4 point calibration range (<1000 ug/ml Isopar). 1,3,5 trimethyl benzene is used as an internal standard for a retention time marker. Eicosane (C-20) is used as in internal standard for quantification purposes. Reporting limits are based on the lowest calibration standard of 40 ug/ml. Naphthas will be identified in the range of 170-270 degrees centigrade hydrocarbon boiling points. If hydrocarbons are found present outside of this B.P. range they will not be reported as naphthas.

2. These details should be documented within an SOP for the QA Plan if practical at this late date.

3. Using a 40 ug/ml standard as a Reporting Limit,

   a D.L. of 500 ug/l will be obtained for water,
   and a D.L. of 17 ug/g,
   (or 17 mg/kg) will be obtained for soil.

   We can not determine the concentrations of standards in Figure 1 of Technical Memorandum. If Naphthas are identified at a response less than 50 ug/ml in final extract, shouldn't these be reported as being present? We can not determine, if 40 ug/ml is a reasonable or ultimate detection limit, especially for the groundwaters. We are confident that detection limits will not be a problem for soils in the area of the Tank Farms.

   The internal standard eicosane (C-20) is used as an internal standard for quantification purposes. A negative bias of 20-30% is obtained for Isopars K,L. & M (see last page of text from November 18, 1989 Technical Memorandum) through use of this internal standard versus external standards. Is eicosane added as a pure compound, or is it part of a hydrocarbon mixture of Figure 1 - Calibration Check Standard labeled "5A - AND"? How accurate is the concentration of this compound known since it affects the concentration of calculated Isopar? Is the response factor different for C-12 and C-20? Will more accurate quantification results be obtained using external standards? The eicosane approach is more cost effective than external standards, on the other hand.

   Summary - Excellent technical approach for the determination of Isopars. It is the best we have reviewed the past two years for RCRA & Superfund sites. A SOP should be provided to document details not readily apparent, however, this may not be needed if it

delays the project. There is a 20-30% bias for quantification of Isopars versus eicosane. Is this significant? Can an alternative be found without causing larger biases?

C. Separate Issue for Naphtha Analysis of Soils by ENSECO/RMAL.

The approach by ENSECO/RMAL for Isopar Naphthas was discussed above. Any SOP or the QA Plan should identify that the Naphthas will be reported on a dry weight basis (103-105 degrees centigrade) for soils. This is an issue for all soil analysis by RMAL, which prefers to report all analyses on a wet weight basis with no % solids determination of soils.

D. Letter of January 19, 1990 from CH2M-Hill concerning EPA Lab Audits of CBC Environmental services.

CH2M-Hill has proposed shifting most general chemistry analyses of surface waters to ENSECO/RMAL. This is acceptable. Dave Payne, Chemist, CRL Region V spoke earlier to Lindsay Brauer of the RMAL laboratory during February 1990. Test procedures used by ENSECO/RMAL are appropriate and acceptable.

General Chemistry - Surface Waters

Table B-2 of the QA Plan should be changed in the following way:

1. RMAL will be using a Hach or OIC Low-Level COD methodology applicable between 5 and 50 mg/l or 5 and 100 mg/l. The system is somewhat proprietary (proprietary to OIC or to Hach Chemical Co) but it works. It does not correspond to any EPA methodology in that the colorimetric read-outs measure the decrease of Cr (VI) color while EPA Method 410.,4 measures the increase of Cr (III) color. The Hach or OIC methodology does not correspond to any of the titrimetric Methods - 410.1, 410.2, or 410.3. The QA Plan should specify "Hach low level COD (colorimetric)" or "OIC low level COD (colorimetric)" depending on source.

2. Total Phosphorus will be done using a Method 365.1 with a 2 reagent system. It works well with a detection limit of 0.01 to 0.02 mg/l.

3. RMAL will be using an Auto Analyzer system for ammonia (Method 350.1). It is appropriate and acceptable.

The test procedures presently cited for TOC, nitrate + nitrite, alkalinity, TSS, and TDS are unchanged. RMAL will be using the test procedures cited.

For CBC's determination of BODs, it is appropriate if BOD values of 5 mg/l or greater are acceptable. CBC determines BOD's as if they are a waste sample through dilutions and seeding. If values below 5 mg/l are not needed for the project, stay with CBC and its procedure as outlined

by CH2M-Hills letter of January 19, 1990 and recommendations for the BOD test provided for the September 1989 lab evaluation by the CRL.

It is appropriate to use ENSECO/RMAL for the general chemistry analysis; however, RMAL and CH2M-Hill should jointly determine the sample bottles/preservatives for these test. We believe RMAL will be providing bottles and preclosed preservatives. QA Plan Table BA-20 should be updated by CH2M-Hill, as appropriate.

It is now apparent that STS will be determining the TOC in soil. Our recommendations for this methodology is that it be consistent with Chapter 29 of "Methods of Soil Analysis".

E. Letter of November 10, 1989 from ENSECO/RMAL to Dave Payne, CRL U.S. EPA Region V concerning Pesticide/PCB Separations.

We previously commented on this letter in our comments of February 15, 1990 (pg. 4 of 14). The subject is the use of silicic acid to separate certain pesticides from PCBs in the 6% ether/hexane fraction from florisil, and is most applicable to the stream sediments (chlordane, DDT's,PCBs) of the Johnson Wax RFI. We have used the silicic acid procedure the past 10 years in the determination of pesticide residues in Great Lakes fish, and found it useful. The subject was also discussed in CH2M-Hill's letter of March 2, 1990. The use of silicic acid is tied also to ENSECO/RMAL not using separate 6%, 15%, and 50% ether fractions from florisil, but using combined extracts for pesticide/PCB analysis.

Our contention is that the silicic acid analysis is not necessary if no PCB's are present. If significant PCB's are present, the silicic acid technique must or should be used in conjunction with the 6% florisil fraction. If sediments are extracted with significant matrix interferences using a combined 6%, 15% and 50% fraction, the separate 6% fraction should have first been done, before we consider use of the silicic acid fraction. If we don't have the 6% fraction, we can not use silicic acid.

We believe the combined extracts can be used so long as detection limit objectives are not degraded and so long as chemical noise does not provide false positive results; however, if extract interferences are present, the 6% florisil fraction will not be available for the determination of most pesticides of Method 8080 and for the determination of PCB's. ENSECO's SOP'S do not provide for the saving of 3 separate fractions but combine all of them into one fraction. Use of silicic acid would require re-extraction of the sample. This may not be practical. If the 6% florisil fraction is available, silicic acid can be used if PCB's are present.

cc: D. Payne, CRL-EPA
G. Schupp, QAS-EPA
E. Evangelista, CRL - EPA
K. Gunter, CRL - EPA

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION V

DATE: APR 26 1990

SUBJECT: Laboratory Evaluation - ENSECO/Rocky Mountain Analytical Laboratory (RMAL), Denver CO for Johnson Wax, WI
RCRA Facility Investigation (RFI)

FROM: Curtis Ross, Director Chuck Elly for
Central Regional Laboratory

TO: William E. Muno, Chief
RCRA Enforcement Branch

ATTN: Glenn Sternard

Attached is the final lab evaluation report for ENSECO/RMAL, Denver, CO. The points discussed are:

1. A restatement or summary of changes in compounds to report for RMAL analyses.

2. Problems (possible) in adding 9 phosphorus pesticides to Method 8270, with a request for justification (promised by RMAL).

3. Further details (as requested by CH2M-Hill in their letter of March 2, 1990) on PQL's used for four compounds of Method 8270, and for water miscible volatiles of Method 8240. New PQL's proposed by RMAL for the 9 phosphorus pesticides appear too large for Method 8270. Recommendations are made for possible changes.

4. Operations of GC Methods 8140, 8150 and 8080 with recommendations for reporting limits. The DDT and endrin degradation checks are discussed, as well as standard preparation(s) for Method 8150, and calibration curves for all of these procedures.

5. Sample extract clean-ups and surrogate and matrix spike compounds. These should be principal topics for any technical meeting. We should find as reasonable an approach as possible.

6. Adequacy/Inadequacy of SOP's for ENSECO/RMAL were discussed in previous February 15, 1990 correspondence from CRL to RMAL. CH2M-Hill in letter of March 2, 1990 said that these deficiencies will be addressed.

7. The question of benzene methodology has been referred to QA Section of Region V, and the CRL and QA Section agree with the recommendations of ENSECO/RMAL and CH2M-Hill.

With the memo reports of April 4 and 20, 1990 to you (concerning non-Appendix IX issues), and with resolution of the above points, we believe ENSECO/RMAL will be appropriate and acceptable for the Johnson Wax RFI. We do not yet have an acceptable alternative to the current extraction solvent system for Method 8150 analysis of soils.

cc: G. Schupp, QA Section-EPA
D. Payne, CRL-EPA
E. Evangelista, CRL-EPA
K. Gunter, CRL-EPA

I. INTRODUCTION

During January and February 1990, the Region V Central Regional Laboratory (CRL) reviewed the analysis records (Appendix IX), for 1 water (field blank), 2 soils, and 2 wastes as provided by ENSECO/RMAL. These were reviewed for conformance with ENSECO's QA Plan SOP's and QC practices, for conformance with SW-846 requirements, and for technical merit. ENSECO/RMAL was able to provide indexes to the raw data records for calibration curves, daily continuing standards, certain blanks, and samples or sample extracts. Questions concerning the data records were FAXED to RMAL on January 16, January 26, January 30, and February 8, 1990. An initial evaluation of ENSECO's Appendix IX procedures was FAXED to RMAL and to CH2M-Hill on February 15, 1990 subsequent to telephone conference calls between the CRL and RMAL. ENSECO/RMAL should be complimented for their cooperation in 1990. RMAL and CH2M-Hill, in a letter dated March 2, 1990 have voiced either their concerns or agreements with items in the February 15th evaluation. This memo report is now a finalized report for ENSECO-RMAL's analysis procedures for appendix IX and associated analyses for the Johnson Wax RFI. This memo report does not address dioxin/dibenzofuran analyses by method 8280 to be done by ENSECO/CAL.

II. APPENDIX IX COMPOUNDS OR PARAMETERS

A. The following compounds should be noted as not being detectable by ENSECO/RMAL using GC/MS methods.

1. 4-Nitroquinoline-1-oxide
2. p-Phenylenediamine
3. Hexachlorophene
4. Pyridine (soils)
5. 1,4 Dioxane at a D.L. of 100 ug/l in water. 1,000 ug/l standards of 1,4 Dioxane did not provide sufficient sensitivity to warrant using a D.L. of 100 ug/l.

6. Ethyl Cyanide at a detection limit of 5.0 ug/l. The first 4 compounds are a fact of life. Many labs can not detect these compound. There is disagreement between the CRL and a RMAL on D.L. for 1,4-Dioxane. The ethyl cyanide was not discussed previously with RMAL.

7. Famphur and Dimethoate.

B. The following compounds should be added to data reports for RFI.

1. Cis-1,2-Dichloroethene (Presently part of RMAL Method-8240 calibration standard)

2. α&γ-chlordane - This can be added to Method 8080 calibration standards based on QA Plan objectives for the site.

C. Fluoride can be deleted from RMAL's list of Appendix IX compounds since it is not part of Appendix IX.

D. Method 8140 Pesticides (Phosphorus Containing)-RMAL-plans to add these 9 phosphorus containing compounds to the list of analyses to be determined by Method 8270. The 9 compounds are currently part of the continuing calibration standard solution used for instrument calibration in Method 8270. Method 8270 has been deemed acceptable and more cost-effective than the more sensitive Method 8140. One method (Method 8140) will then be eliminated for Appendix IX determinations.

Two of the 9^compounds are believed to be non-detectable by method 8270:

1. Dimethorate - The high pH of the initial BNA extraction can destroy this compound, based on U.S EPA method validation studies. See June 5, 1987, report copy provided to ENSECO/RMAL

2. Famphur - GC/MS response appears to be as bad as hexachlorophene; however, its GC response for Method 8140 is not very good based on data package review.

Since writing the February 15, 1990, initial lab evaluation report, the CRL has spoken with different Appendix IX laboratories. Certain lab personnel are concerned about the high pH values in the BNA water extraction for the phosphorus pesticides of Method 8140. The June 5, 1987, method validation of EPA for the extraction of the 9 compounds used a seperatory funnel extraction for the waters. RMAL is using a continuous liquid-liquid extraction of 16 hours at high pH. RMAL has indicated, during telephone conference calls, that MDL studies have been done by Method 8270 for the 9 compounds.

1. Recommendation:

RMAL should provide recovery data for BNA extraction of the 9 compounds using the continuous liquid extractor. This is a new comment from Region V, but it is reasonable. The initial extraction should be done at a high pH value (if this has never been done). RMAL should justify recovery of these compounds by Method 8270 while Method 8140 has only a neutral pH extraction of waters. In a telephone conversation with RMAL on 4-3-90, RMAL personnel promised to provide these recovery data for the 9 pesticides of Method 8140.

benzene				
Sym-Trinito-benzene	50	10	5,300	0.06
Demethoate	50 **	Not Det.*	21,000	0.31-0.39
Disulfton	50**	50*	43,000	0.52-0.62
Parathion	50**	50*	14,000	0.17-0.21
Famphur	50**	100*	No Response	0.0
Methyl Parathion	50**	50*	17,000	0.24-0.28
Phonate (Thimet)	50**	100*	15,000	0.18-0.20
Sulfotepp	50**	50*	15,000	0.12-0.16
Thionazin	50**	50*	14,000	0.26-0.31
0,0,0-etc.	50**	50*	1,000	0.13-0.16

* - Data Faxed to CRL on 2-14-90
** - 1989 Data may not be applicable to 1990.

Review of the 1989 data package showed RMAL to report the presence and concentration of all MS "hits" even if the concentration was less than the "PQL". Data reduction would then change these values to "less that PQL" even though acceptable mass spectra are present (The CLP will report these low concentrations with a "J" qualifier).

The above data for Method 8240 water soluble-low purge efficiency volatiles show no consistency between area counts of mass spectra quant ion, PQL used, and response factors. Area counts for good acting compounds with response factors greater than 0.30 or 1.0 should certainly be detected below their PQL of 5 or 10 ug/l. Also, why does acetone have a PQL of 25 ug/l while acrolein is reported using a PQL of 100 ug/l and 1,4 Dioxane is reported with a PQL of 100 ug/l?

For Method 8270, is it reasonable to expect a PQL of 10 ug/l is appropriate for sym. trinitrobenzene? Why are the PQL's set at 50 or 100 ug/l for the 9 Method 8140 pesticides when their response factors are significantly larger than 0.10 or 0.20? By changing from Method 8140, PQL's are increased 100 fold through use of Method 8270.

Recommendations:

1. Report all positive MS "hits" with acceptable mass spectra as recommended by CH2M-Hill in their letter of March 2, 1990.

2. Justify the large PQL 's for 7 of the 9 Method 8140 pesticides. Whey don't they approximate 10 ug/l?

3. Significantly increase the PQL for 1,4 dioxane and Ethyl Cyanide.

4. Justify why Daily Std concentrations are lower than their PQL's for Acrolein, and Acrylonitrile. How can you calibrate or

update an instrument using concentrations smaller than their reporting limits?

5. Is there a rationale for selection of PQL's for the water soluble compounds of Method 8240?

6. Why is the PQL set at 10 ug/l for sym. trinitrobenzene?

7. Justify whey Famphur is reported at all. Does RMAL have extraction recovery data for Dimethoate to justify its non-detection other than the EPA validation report?

IV. GC INSTRUMENT OPERATIONS - METHODS 8080, 8140 AND 8150

The instrumental operations for ENSECO Methods 8080, 8140, and 8140 are fairly straight forward, after review of a "typical data set". Five point calibrations are provided for the CLP and TCL pesticides of Method 8080. Single standard solutions are injected for chlorobenzilate, isodrin, kepone, and the multicomponent Arcolors, tech. chlordane, and toxaphene. External standards are used to quantify these compounds in sample extracts. ENSECO is to be complimented on their thorough documentation of Rention Time windows and their use in their Nelson Data System for the CLP TCL pesticides.

1. Recommendation:

   ENSECO needs, as a reference, 5 point calibration curves for the determination of linearity, acceptable instrument response, detection limits, etc., for the 3 Appendix IX pesticides, α-chlordane, α-chlordane, and at least 4 of the routinely determined Acrocolors (1242, 1248, 1254, and 1260). We would encourage use of the Nelson Data System for these latter compounds, but do not require it. It is helpful to use the data system to plot the reference calibration curves, instead of reviewing individual calibration factors.

2. Recommendation:

   ENSECO should consider that either DDT or endrin degradation must be <20% when pesticides are reported. We would encourage these degradations to be smaller. We realize that automated instrument operation does not allow these determination to be made prior to sample analysis; however, SW-846 Method 8080 mandates the degradation be less than 20% for either compound.

The instrument operations for Method 8140 were routine; however, 3 of the 9 compounds were inappropriate concentrations for their 5 point calibration curves. This is documented in case the methodology is used for other projects.

ENSECO's Method 8150 had a 5 point calibration curve, of excellent

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
230 SOUTH DEARBORN ST.
CHICAGO, ILLINOIS 60604

REPLY TO THE ATTENTION OF:

JUL 18 1989

5SCRL

Jerry Parr, Technical Director
ENSECO-RMAL
4955 Yarrow Street
Arvada, CO 80002

To Jerry Parr
From: Dave Payne
~~The~~ We inadvertently left-out one set of parameters - see bdy

Dear Mr. Parr:

We are requesting information concerning analytical methodologies and QC procedures to be used by three ENSECO laboratories for RCRA Facility Investigations (RFI's) planned at 5 sites within Region V. Detailed information is requested so that we will not need to make on-site laboratory evaluations requested by our Region V RCRA Enforcement Branch. Acceptable responses by a laboratory should constitute a laboratory evaluation. David A. Payne, Chemist, of our staff spoke with you the week of July 3, 1989, so this letter is not unexpected. The planned, or ongoing, RCRA RFI's are

1. S.C. Johnson, WI

   Consulting Engineering Firm: CH_2M-Hill
   Parameters: Appendix IX - RMAL
   Other Organics - RMAL
   (Method 8010/8020, Specialized Method 8080)
   Naphtha - RMAL
   Dioxins/Dibenzofurans - Cal Analytical
   Sample Types: Groundwater, Soils, Surface Water, Sediments

2. Freeman Chemical, WI

   Consulting Engineering Firm: Hatcher/Sayre Inc.
   Parameters: Appendix IX - ENSECO/ERCO
   (Do not know if Method 8280 is to be done at this time)
   Sample Types: Groundwater

3. Stanley Tools, MI

   Consulting Engineering Firm: Dames & Moore, Cincinnati, OH
   Parameters: Appendix IX - RMAL (Includes Method 8280)
   Sample Types: Groundwater, Soils

Also needs "priority pollutants"

g. MS/MSD results (if any)
h. Order of all samples, calibrations checks, blanks, QC audits, etc.
i. Quant Reports and Calibration Files for the above measurements.
j. We do not need to obtain sample analysis results except for associated surrogates, internal standard, and MS results.

For the above set of analysis records we would expect the complete library (Finnigan nomenclature) or I.D. File (HP nomenclature), calibration files for associated 5 point calibration curves, and Quant Report(s) used to generate I.D. Files.

C. For GC analyses (Methods 8080, 8140, 8150, and specialized 601/602 or 8010/8020 procedures and Method 8120 for C-Series compounds, if available) we request a "CLP type" data package for each method that provides the same type of information in Item #B above except we will expect chromatograph copies for each of the primary and secondary columns used, any column performance or degradation checks, and Data Files with chromatographs and quantitations. We would expect Method Files, 5 point calibration curves with chromatographs associated with the above sample analysis sets.

D. For Method 8280, we expect to review a typical evidential file, with purging of sample analysis results as necessary to protect client confidentiality. We are uncertain yet if one, or 3 ENSECO labs are to provide this type of analysis for dioxins/dibenzofurans. This needs to be discussed further.

E. For inorganics, we request you provide records for a recent, typical set of Appendix IX analyses that includes "CLP type" QC summaries with sample handling records, prep logs, bench records of analysis showing the order of calibrations, QC checks, sample analysis and their results for ICP emission measurements, the 4 graphite furnace metal determinations, cyanide, and sulfide. We request interelemental correction and background correction information for each ICP emission spectrometer instrument being used.

F. We have received an ENSECO QA Program Plan for the SC Johnson site. Is this applicable to the remaining sites? Please provide applicable QA Program Plans to the remaining sites as appropriate.

G. We request that an example data deliverable package for the above analyses be provided to us from each applicable laboratory. It should include formats for sample results (soils and waters), detection limits, blanks, QC information, etc. We understand the laboratory QA Program Plans will define detection limits and reporting of blank determinations.

Questions concerning the above information should be directed to David A. Payne, Chemist at (312) 886-1970.

Recent conversations with Occidental Chemical Corp. indicated RMAL provided Appendix IX determinations for a few leachates from the company's Michigan site. The analyses were done 6 months ago. Analysis records for these determinations could be a useful typical data set to provide the requested information so long as it is representative of future Appendix IX analyses. Client confidentiality need not be protected. Occidental Chemical Corp. wants us to review these data. Contact Dr. Alan F. Weston Oxy Chem, Niagara Falls, NY at (716) 286-3607 concerning this request.

During August 1987, Mr. Payne of our laboratory will be requesting information on special topics for the RFI projects. Two items to consider are

1. One RFI has past sediment data with DDT compounds, chlordane, and PCB's all present at once. Does RMAL have appropriate clean-up/separation procedures in place for sediment extracts from this site?

2. Past data from one site shows the presence of 10,000 ug/l or more toluene in certain groundwaters. Can Appendix IX volatile determinations be made with any meaningful detection limits in the presence of a single large volatile compound concentration?

Sincerely yours,

Charles J. Elly for

Curtis Ross
Director

c.c. W. Muno, EPA
L. Lodisio, EPA
R. Smith, EPA
G. Sternard, EPA
B. Strom, EPA
E. Helmer, EPA

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-3
SOP Number: LM-RMA-2037
Title: Metals Analysis: Inductively Coupled Plasma

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

Section 8.1: Sample preparation SOPs LM-RMA-2002 and LM-RMA-2029 referenced from this section are located in QAPjP Appendix B-4A.

Section 8.4.1: The concentration of metals in the DCS QC samples will be documented in the project reports. Concentration of metals in the calibration standards will be available in the raw data. (Also see Section 6 of this SOP.)

Section 9.2.9.1: Matrix spike levels and acceptance control limits are provided in QAPjP Appendix A-11.

SOP Appendix 1: Re: Arsenic, Selenium and Thallium. Enseco method detection limits and reporting limits as stated as stated in this appendix are correct, but are high relative to typical DQOs. However by reference to QAPjP Section IX it can be seen that these analytes will be evaluated by GFAA methods, and each has a reporting limit of 0.005 mg/l as per QAPjP Appendix A-10.

ICP Interelement Interference: Information on interelement intereference, noted in Section 3.3.2 of the SOP, has been added to QAPjP Appendix B-3. IEC composition is subject to change by lot number. Should a different lot number be in use at the time of analysis, additional information concerning the the composition and concentration of the standard solution used for interelement correction will be provided with the data report. (Also see Sections 3.1 and 6 of this SOP.)

2700 Chandler Avenue, Building C
Las Vegas, Nevada
89120

702 795-0515

ICF TECHNOLOGY INCORPORATED

October 9, 1990

INTERFERENCE CHECK SAMPLE SET - PART A (0590) WITH PART B (1089)

The interference check sample set is to be used to verify interelement and background correction factors of inductively-coupled plasma spectrometers. This reference material consists of two concentrated solutions. The Part A (0590) solution contains the four interferent elements: Al, Ca, Fe and Mg. The Part B (1089) solution contains the analytes: Ag, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, V and Zn.

The "True Value" concentrations of the elements are listed in Table 1. These are the concentrations that result when the solutions are prepared and analyzed according to the following instructions:

Part A (0590), Inferferents: Pipet 10 mL of the Part A solution into a 100-mL volumetric flask and dilute to volume with 2% v/v HNO_3. Analyze this solution, Soln. A, by ICP-OES.

Part B (1089), Analytes, mixed with Part A (0590), Interferents: Pipet 10 mL of the Part A solution and 10 mL of the Part B solution into a 100-mL volumetric flask and dilute to volume with 2% v/v HNO_3. Analyze this solution, Soln. AB, by ICP-OES.

The ICS Part A and B solutions were prepared from nitrate salts. The solutions are stable at room temperature and should not be frozen. To insure the stability of silver, which is contained in the Part B solution, the solution-set should be stored in the dark.

The ICS set was prepared for contract work under the Inorganic Statement of Work, SOW 787. However, for work under older contracts, mix Parts A (0590) and B (1089) as specified in these instructions to produce Soln AB. This solution is similar to the ICS X-85 and ICS X-85 B solutions, which are no longer available.

The ICS Part A and B solutions were prepared under contract to the EPA. If there are any questions regarding this reference material, please call (702) 795-0515.

Table 1. "TRUE VALUE" CONCENTRATIONS FOR THE ELEMENTS IN INTERFERENCE CHECK SAMPLE PART A (0590) MIXED WITH PART B (1089)

Element	Concentration (ug/L)	
	Soln. A	Soln. AB
Al	540055	536472
Ba		502
Be		480
Cd		907
Ca	494040	512228
Cr	21	529
Co		477
Cu		543
Fe	206236	199845
Pb		4724
Mg	531358	527530
Mn	34	496
Ni		940
Ag		960
V		509
Zn	216	1208

Subject or Title: Metals Analysis: Inductively Coupled Plasma (ICP) (Industrial Metals)

SOP No.:	Revision No.:	Effective Date:
LM-RMA-2037	1.0	July 1, 1991

Supersedes: Original (February 1, 1990)

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1. Scope and Application

1.1 Analytes

This method is applicable to the determination of trace metals using Enseco-RMAL Jarrell-Ash ICAP 61 instrumentation. See Appendix 1 for specific analyte list which is subject to change.

1.2 Applicable Matrices

This method is applicable to a variety of environmental matrices including dissolved, recoverable and total water samples, as well as digests of soils, wastes, and leachates.

Prepared by: Fred Velasquez	Date: July 1, 1991
Management Approval: David B Roberts	Date: 8/12/91
QA Officer Approval: Bill Hayes	Date: 08/07/91

1.3 Reporting Limits

Standard Enseco reporting limits range from 0.002 to 5 mg/L in aqueous samples. Soil, waste and leachate limits are 10-100x higher. All limits are subject to elevation upon dilution due to analytes exceeding linear range of specified instrument. Method detection limits (MDL's) are also available. Consult Appendix 1 for specific analyte information which is subject to change annually.

1.4 Dynamic Range

The dynamic range is element specific. For most analytes it extends to 100 mg/L in solution. Higher concentrations may be analyzed by dilution of the sample. Consult Appendix 1 for specific analyte information.

1.5 Analysis Time

Approximately 40 samples can be analyzed per instrument-shift. This includes all QC samples and entry into the LIMS system. Sample preparation time is not included.

2. Method Summary

2.1 Aqueous and digested samples are nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the quartz torch and into the plasma. The plasma of ionized argon is sustained at a temperature as high as 10,000^{o}K by a Radio Frequency (RF) generation source.

2.2 Once the aerosol reaches the plasma, the sample is desolated (decomposed), the elements are excited and emit radiation at their characteristic wavelength. See Appendix 6.

2.3 The emitted light energy is then directed through the exit slits and projected on photo multiplier tubes (PMT) specific for each element. The PMT's convert the light energy to an electrical signal that is digitized and processed by the instrument's computer.

3. Comments

3.1 Interferences

3.1.1 Background emission may be caused by, among other things, continuing emission and viscosity differences. Background

correction points are required for each element. (See ICAP 61 operator's manual).

3.1.2 Interelement interferences occur when elements in the sample emit radiation and wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter Element Corrections (IEC's) must be applied to the analyte to remove the effects of these unwanted emissions. To calculate an IEC, standardize the ICP, set analysis made to Standardize intensity ratio and analyze the "interfering element" at its upper linear range. Divide the concentration of the analyte by the concentration of the "interfering element." That number is the value entered in the IEC for the analyte.

3.2 Helpful Hints

3.2.1 Use correct digest for ICP "HCL/HNO_3" green sticker.

3.2.2 When not using a print out, draw one line through printout diagonally. Write not used, initial, and date.

3.2.3 Use correct ICP Method (SW-846 #6010), run dilutions immediately after the sample if running manually or at the end of the run if using an autosampler.

3.2.4 Saturated Element

3.2.4.1 Zinc, when saturated aspirate with 10% HNO_3 acid.

3.2.4.2 Cadmium, when saturated aspirate with 10% HNO_3 acid.

3.2.4.3 Molybdenum, when saturated aspirate with 10% HCL acid.

3.2.4.4 Calcium, when saturated aspirate with 10% HNO_3 acid.

3.2.4.5 Chromium, when saturated aspirate with 10% HCL acid.

3.2.4.6 Iron, when saturated aspirate with 10% HCL acid.

3.2.4.7 Sodium, when saturated aspirate with 10% HNO_3 acid.

3.2.4.8 Barium, when saturated aspirate with 10% HNO_3 acid.

3.2.4.9 Boron, when saturated aspirate with 10% H_2SO_4 acid.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco-Rocky Mountain Analytical Laboratory Safety Plan. Lab coats and safety glasses are required in all laboratory area at all times. If you have any questions or safety concerns, see your supervisor or safety officer.

4.2 Many of the metals determined by this method are extremely toxic and/or carcinogenic. Care should be taken when preparing standards from concentrated stock solutions. Wear safety glasses, gloves, and lab coat.

4.3 All of the samples may contain unknown contaminants at unknown levels other than the analytes being tested. Appropriate care should be taken when handling samples.

4.4 The plasma emits strong UV light and is very harmful to vision. AVOID LOOKING DIRECTLY AT THE PLASMA.

5. Apparatus

5.1 Thermo Jerrell Ash ICAP 61 Simultaneous Plasma Emission Spectrometer which consists of a 0.75 meter polychromator that accepts up to 63 channels, an RF generator, an inductively coupled argon plasma (ICAP) excitation source and an IBM compatible data acquisition system.

5.2 Rainin Rabbit Peristaltic Pump

5.3 Eppendorf 10-100 uL and 100-1000 uL adjustable pipettors

5.4 Oxford 1-5 mL and 5-10 mL adjustable pipettors

6. Reagents, Standards, and Supplies

6.1 Calibration standards prepared monthly by Environmental Resource Associates. These are verified against EPA standards before use.

6.2 ICV concentrate solutions are prepared by EPA Las Vegas/UNLV. Working solutions are prepared daily according to the dilution instructions accompanying the concentrates.

6.3 Interference check solutions A and B are prepared by EPA Las Vegas.

6.4 IND CCV SOLN's 1-3 are prepared by Inorganic Ventures and diluted and run daily. Verified against EPA ICV standards before use.

6.5 Approximately 8 ppm Ba profiling solution prepared at RMAL.

6.6 1000 ppm yttrium stock solution

6.7 Ind. ICP analytical spike A, B, C prepared by the standards prep analyst. Verified against EPA standards before use.

6.8 Five dram snap cap vials

6.9 Disposable .45 um syringe filters and syringes (Gelman or equivalent).

7. Sample Collection ,Preservation and Holding Times

7.1 See SOP #LP-RMA-0056 "Bottle Prep and Cooler Shipping"

8. Procedure

8.1 Preparation

8.1.1 Consult SOP's; LM-RMA-2002 Total,Recoverable,Dissolved, and Total Metal:Acid Digestion for Water, EP TOX and TCLP Extracts.
LM-RMA-2029 Total Metals Digestion Preparation.: Acid Digestion for Soil/Waste samples.

8.2 Instrument Set-up

8.2.1 Turn on coolant water, argon, and exhaust fan.

8.2.2 Confirm that the argon supply is sufficient.

8.2.3 Turn computer, printer on.

8.2.4 Turn mass flow controller on.

8.2.4.1 Channel 3 should be set at 600 and read 600 ± 10. Channel 4 should set and read 15.

8.2.5 All switches on gas flow panel should be on or in up position.

8.2.6 Turn on peristaltic pump and introduce DI H_2O to torch for approximately 1 minute.

8.2.7 Push RF button on.

8.2.8 Turn power knob up to 75-100 (bottom of yellow on meter).

8.2.9 Turn sample switch off to stop H_2O from entering the torch. (Make sure auxiliary torch switches are on.)

8.2.10 Press ignition button while turning power knob up slowly all the way. Torch should be on, if not stop and try again.

8.2.11 Turn sample switch on while turning auxiliary switch off at same time, slowly.

8.2.12 Aspirate 1000 ppm yttrium. The torch will be blue and pink. Check the plasma. The pink and blue should be highly visible. The work coil should sit on the Y line of the polychrometer. The blue and pink should meet one line below the X line. See Appendix 7.

8.3 Instrument Profiling

8.3.1 Aspirate the 8 ppm Ba profiling solution

8.3.2 From the analysis screen on the computer, press the appropriate function keys to initiate manual instrument profile. Follow the step by step instructions that appear on

the computer monitor. (Note: The directions given for turning the micrometer are backwards so turn the micrometer in the opposite direction.) Hit the F-9 key to keep your profile.

8.4 Instrument Calibration

8.4.1 From the analysis screen press the calibration key. The calibration screen will appear on the monitor. Aspirate ERA calibration standard #1 (blk). Make sure the standard is in the torch before telling to monitor to expose. (20 seconds). Hit F-1 to initiate exposure of standard calibration #1. Wait until the results have printed before pulling it out. Rinse with DI HNO_3 for approximate one minute. Hit the F-9 key to keep standard #1. Continue this procedure with the rest of the calibration standards in the order which they appear on the monitor. Make sure you F-9 (done/keep) after each standard. F-9 to keep all standards. The stop/intercept screen should appear. Hit F-2 to print screen. F-9 your way out to operation screen and hit enter. You are now at the analysis screen.

8.5 Sample QC Set-up

8.5.1 An ICV-1/3, ICS-A/B, Ind. CCV must be made up daily from the concentrate solutions.

8.5.1.1 EPA ICV-1/3: Aliquot 1 mL of ICV-1 and one mL of ICV-3 in a 30 dram vial. Bring to 10 mLs with 8 mLs of 1% HNO_3.

8.5.1.2 EPA-ICS-A/B: Aliquot 1 mL of EPA ICS-A and 1 mL EPA ICS-B into a 30 dram vial and add 8 mLs of 1% HNO_3.

8.5.1.3 Ind. CCV: Use 0.2 mLs of each Inorganic Ventures solutions CCV-1, CCV-2, CCV-3 in a 30 dram vial. Add 19.4 mLs of 10% HCl.

8.6 Data Package Set-up

8.6.1 Find the project or projects you will be working on.

8.6.1.1 If running an aqueous total, recoverable, soil/waste, EP-1, EP-2, or TCLP use the QC lot number from the bottle. (These are digested samples.)

8.6.1.2 If you are running an aqueous dissolved, use the date of analysis for QC lot number.

8.6.2 Write the project number and sample numbers on the ICP cover sheet. Write the project QC lot number (see 8.5.1.1/8.5.1.2). Write the analysis test (see daily JAK list). For special instructions go into LIMS. Type LIMSMENU and hit the enter key. Project/Data Maintenance should be highlighted. Hit the enter key, go to Group Code and hit the enter key, type the project number and hit the enter key. This screen will let you know if there are any special instructions. If no special instructions mark the N column on the cover sheet. If there are special instructions hit F-5 (Test List), hit F-5 (More Functions), hit F-7 (Group Instructions). The Group Instructions should appear on the screen. Print the special instructions by hitting the Ctrl key and the P key simultaneously. Mark the Y column on the cover page. If instructions appear but are not applicable to ICP metals, mark Y/NA column. Instructions need not be printed.

8.6.3 Hit the F-10 key, go down to Test Request by sample, and hit the enter key. Type the project number and hit the enter key. Choose correct sample number and find the correct ICP test. Hit F-1 (Result Detail) and hit F-1 (Add/Edit Results). This screen will show you the analytes you will need. Circle analytes on the cover sheet. Hit the F-10 key. The monitor will ask you if you wish to cancel the operation. Hit the "Y" key for yes. Get a "Data Review Program Level 1 Review Checklist", and "ICP DCS Sheet" for each QC lot number, and a "Comment Anomaly Form." The Data Review Checklist is to be filled out before handing the package to the supervisor for reviewing. The DCS sheets should be filled out after running each DCS. The anomaly form should be filled out when anything out of the norm is seen, e.g. dilution, viscosity, color, odor.

8.7 Sample Analysis

8.7.1 From the ICP monitor screen hit F-1. This should bring you to the "Sample Information Screen." Type the QC name or project and sample number under "Sample Name", hit the enter key.

This will bring you to the "Comment" section. Type the QC number and/or any pertinent information that is needed. (Note: No DCS lot number for QC.)

8.7.2 Hit the enter key, this will bring you to the "Operator ID." Type your initials. Hit the enter key. This will bring you to the "Corr. Factor." Type your dilution factor, example: 1X for H_2O and 100X for soil/waste and any other dilution factor that may be required. Aspirate the sample for 20 seconds and hit the F-1 key to start exposure. Wait until second exposure is complete before rinsing in DI H_2O. When the sample is done printing, repeat procedure for next sample. (Note: See QC requirements before running ICV, CCV, DCS, ICB, and CCB.)

9. QA/QC Requirements

9.1 QC Order

9.1.1 ICV-1/3

9.1.2 ICS-A/B

9.1.3 CCV

9.1.4 ICB

9.1.5 Prep Blank (if a digited project)

9.1.6 DCS-1

9.1.7 DCS-2

9.1.8 Seven samples

9.1.9 CCB

9.1.10 CCV

9.1.11 Ten samples, etc.

9.1.12 CCB

9.1.13 CCV

9.2 QA/QC Explanations

9.2.1 Initial Calibration Verification 1/3

9.2.1.1 The ICV-1/3 must be within 10% of the true value. (See Appendix 2.) To make the ICV, see Section 8.5.1.1. The 1 ICV-1/3 is used as an independent source to verify that the ICP is correctly calibrated. The ICV-1/3 is also used to verify standards made at RMAL.

9.2.2 Interference Check Solution A/B

9.2.2.1 The ICS-A/B must be within 20% of true value. (See Appendix 3.) To make ICS-A/B, see Section 8.5.1.2. The ICS-A/B is used to check interelement corrections.

9.2.3 Continuing Calibration Verification

9.2.3.1 In the CCV all elements that are needed must be within 10%. (See Appendix 5.) The CCV is used to verify that the instrument is still in calibration.

9.2.4 Initial Calibration Blank

9.2.4.1 The ICB must be less than Enseco reporting limit for elements being used. (See Appendix 1.) The ICB is used to check for contamination and carryover. Use the ERA calibration standard 1 for ICB's and CCB's.

9.2.5 Prep Blank

9.2.5.1 The prep blank must be less than the Enseco reporting limit and for elements being used. (See Appendix 1.) The prep blank is used to show if any contamination was introduced during the digestion process. The prep blank showing contamination can be used if contamination does not exceed two times the Enseco reporting limit at which time an anomaly must be included with data.

9.2.6 Duplicate Control Sample

9.2.6.1 The DCS's must be within 25% of true value. (See Appendix 4.) The DCS is used to verify that the samples were properly digested.

9.2.7 Continuing Calibration Blank

9.2.7.1 Same as ICB. See Section 9.2.4.1.

9.2.8 Sample Duplicates

9.2.8.1 Duplicates are to be run only when a certain project specifically asks for it. There is no acceptance criteria for project specific QC. If bad RPD, please inform your supervisor.

9.2.8.2 The sample duplicate is run to show reproducibility.

9.2.9 Sample Matrix Spike

9.2.9.1 Matrix spikes are to be run one in every 20 samples. The matrix spike is used to show if there are any interferences in the sample and also to see if you are losing anything during the prep.

9.2.10 Sample Matrix Spike Duplicate

9.2.10.1 Matrix spike duplicates are to be run one in every 20 samples. The MSD is to show reproducibility with matrix interferences.

9.3 Corrective Actions

9.3.1 Follow the corrective actions outlined in the current Enseco QAPP Manual.

9.3.2 If the prep blank for any element being used is more than two times the reporting limit the supervisor/coordinator should be notified.

10. Calculations

10.1 See ICAP 61 Operators Manual

11. Reporting Requirements

11.1 Units

11.1.1 All units will be reported in mg/L or mg/kg. Unless otherwise specified by client.

11.2 Limits

11.2.1 If there is no blank contamination or dilution required report ND for anything reading less than the reporting limit. See Appendix 1.

11.2.2 If the blank shows contamination, (but is less than two times the reporting limit), the reporting limit is raised to the blank value.

11.2.3 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor.

11.3 Significant Figures

11.3.1 Reporting Limits

Results will be reported to two significant figures if less than ten and three significant figures if more than ten. (When entering into LIMS, enter all significant figures up to 5, LIMS will automatically round to appropriate amount of significant figures.)

11.4 LIMS Data Entry

11.4.1 For manual entry refer to Enseco QAPP.

11.4.2 For auto entry refer to Enseco Incorporated Automated Data Upload Program for Instrument Results. Revision 2.0 July 1989.

11.5 Anomalies

11.5.1 Anything that does not meet normal standards. Some examples are: high viscosity dilutions due to interferences, high concentrations, color, and blank contamination. These must be documented and added to the data package.

12. Review Requirements

12.1 See SOP #LP-RMA-0064 "Metals Data Review for Industrial Inorganics."

13. References

13.1 ICAP 61 Operators Manual-Thermo Jarrell Ash corporation, January, 1988 Copyright 1987.

13.2 Test Methods for Evaluating Solid Waste. USEPA SW-846 Third Edition 1986.

13.3 Methods for Chemical Analysis of Water and Wastes. United States Environmental Protection Agency. EPA-600/4-79-020 Revised March 1983.

13.4 Enseco QAPP Manual

13.5 Enseco Incorporated Automated Data Upload Program for Instrument Results Revision 2.0 July 1989.

13.6 M-EQA-0002 Internal QC Checks-Laboratory Performance QC

13.7 Enseco SOP #LM-RMA-2029 "Metals Prep Digestion for Soils"

13.8 Enseco SOP #LM-RMA-2002 "Metals Prep Digestion for Aqueous"

13.9 Enseco SOP #LP-RMA-0064 "Metals Data Review for Industrial Inorganics"

13.10 Enseco SOP #LP-RMA-0056 "Bottle Prep and Cooler Shipping"

13.11 Enseco Safety Manual

APPENDIX 1

INDUSTRIAL INORGANIC CHEMISTRY
METALS DEPARTMENT

ICP INSTRUMENT DETECTION LIMITS - MARCH 1991

Element	MDL* (mg/L)	Enseco Reporting Limit	ILL* (mg/L)
Aluminum	0.009	0.1	500
Antimony	0.02	0.06	100
Barium	0.003	0.01	100
Beryllium	0.002	0.002	40
Cadmium	0.003	0.005	100
Calcium	0.03	0.2	500
Chromium	0.006	0.01	100
Cobalt	0.004	0.01	100
Copper	0.004	0.02	100
Iron	0.007	0.1	500
Lead	0.01	0.05	100
Magnesium	0.008	0.2	500
Manganese	0.003	0.01	100
Molybdenum	0.005	0.02	100
Nickel	0.004	0.04	100
Potassium	0.2	5	500
Silver	0.002	0.01	100
Sodium	0.1	5	800
Vanadium	0.004	0.01	100
Zinc	0.002	0.02	100

Additional analytes and levels available

Arsenic	0.02	0.1	100
Boron	0.005	0.1	100
Lithium	0.002	0.05	500
Phosphorus	0.09	0.3	400
Selenium	0.05	0.2	100
Silica as SiO2	0.2	0.5	500
Strontium	0.02	0.05	100
Thallium	0.5	2	100
Tin	0.03	0.1	100
Titanium	0.003	0.005	100

* MDL = Method Detection Limit (subject to change)
ILL = Instrument Linear Limit

APPENDIX 2

ICV-1 (0489) (after 10-fold dilution)

Element	Value (ug/L)
Al	2010
Ba	1960
Be	483
Cd	502
Ca	48700
Cr	510
Co	502
Cu	515
Fe	2040
Pb	4800
Mg	24600
Mn	504
Ni	480
K	49000
Ag	500
Na	49600
V	488
Zn	3100

ICV-2 (0989): (after 10-fold dilution)

Element	Value (ug/L)
As	52.6
Se	52.6

ICV-3 (0189): (after 10-fold dilution)

Element	Value (ug/L)
Sb	978

ICV-4 (0389): (after 10-fold dilution)

Element	Value (ug/L)
Cd	95.8
Pb	97.5
Ag	98.6
Tl	97.0

All ICV's subject to change due to different lots.

APPENDIX 3

This material was prepared by the University of Nevada, Las Vegas, Quality Assurance Laboratory. If there are any questions regarding this reference material, please call (702) 739-3142.

TABLE 1. "TRUE VALUE" CONCENTRATIONS FOR THE ELEMENTS IN INTERFERENCE CHECK SAMPLE PART A (1287) MIXED WITH PART B (0387)

Element	Concentration ug/L	
	Soln A	Soln AB
Al	511000	508000
Ba		483
Be		474
Cd		909
Ca	476000	470000
Cr	48	513
Co		478
Cu		534
Fe	219000	211000
Pb		4850
Mg	513000	513000
Mn		470
Ni		916
Ag		993
V		475
Zn		973

Solutions subject to change due to different lots

APPENDIX 4

Industrial Metals

DCS Values

Enseco DCS (Duplicate Control Samples)

All values are in mg/L	Concentration
Aluminum (Al)	2.0
Antimony (Sb)	0.5
Arsenic (As)	0.5
Barium (Ba)	2.0
Beryllium (Be)	0.05
Cadmium (Cd)	0.05
Calcium (Ca)	100
Chromium (Cr)	0.2
Cobalt (Co)	0.5
Copper (Cu)	0.25
Iron (Fe)	1.0
Lead (Pb)	0.5
Magnesium (Mg)	50
Manganese (Mn)	0.5
Nickel (Ni)	0.5
Potassium (K)	100
Silver (Ag)	0.05
Sodium (Na)	100
Vanadium (V)	0.5
Zinc (Zn)	0.5

For soil values multiply by a factor of 100.

For leachate values multiply by a factor of 10.

APPENDIX 5

INDUSTRIAL CCV LEVELS

Aluminum	1.0	ppm
Antimony	1.0	ppm
Arsenic	1.0	ppm
Barium	1.0	ppm
Beryllium	1.0	ppm
Boron	1.0	ppm
Cadmium	1.0	ppm
Calcium "Lo"	10.0	ppm
Calcium "Hi"	--	ppm
Chromium	1.0	ppm
Cobalt	1.0	ppm
Copper	1.0	ppm
Iron	1.0	ppm
Lead	1.0	ppm
Lithium	1.0	ppm
Magnesium "Lo"	10	ppm
Magnesium "Hi"	--	ppm
Manganese	1.0	ppm
Molybdenum	1.0	ppm
Nickel	1.0	ppm
Phosphorus	1.0	ppm
Potassium	10	ppm
Selenium	1.0	ppm
Silica	21.4	ppm
Silver	2.0	ppm
Sodium	10	ppm
Strontium	1.0	ppm
Tin	10	ppm
Thallium	10	ppm
Titanium	1.0	ppm
Vanadium	1.0	ppm
Zinc	1.0	ppm

Inorganic Ventures CCV-1 Lot #G-MEB0439
Inorganic Ventures CCV-2 Lot #G-MEB05007
Inorganic Ventures CCV-3 Lot #G-MEB04041

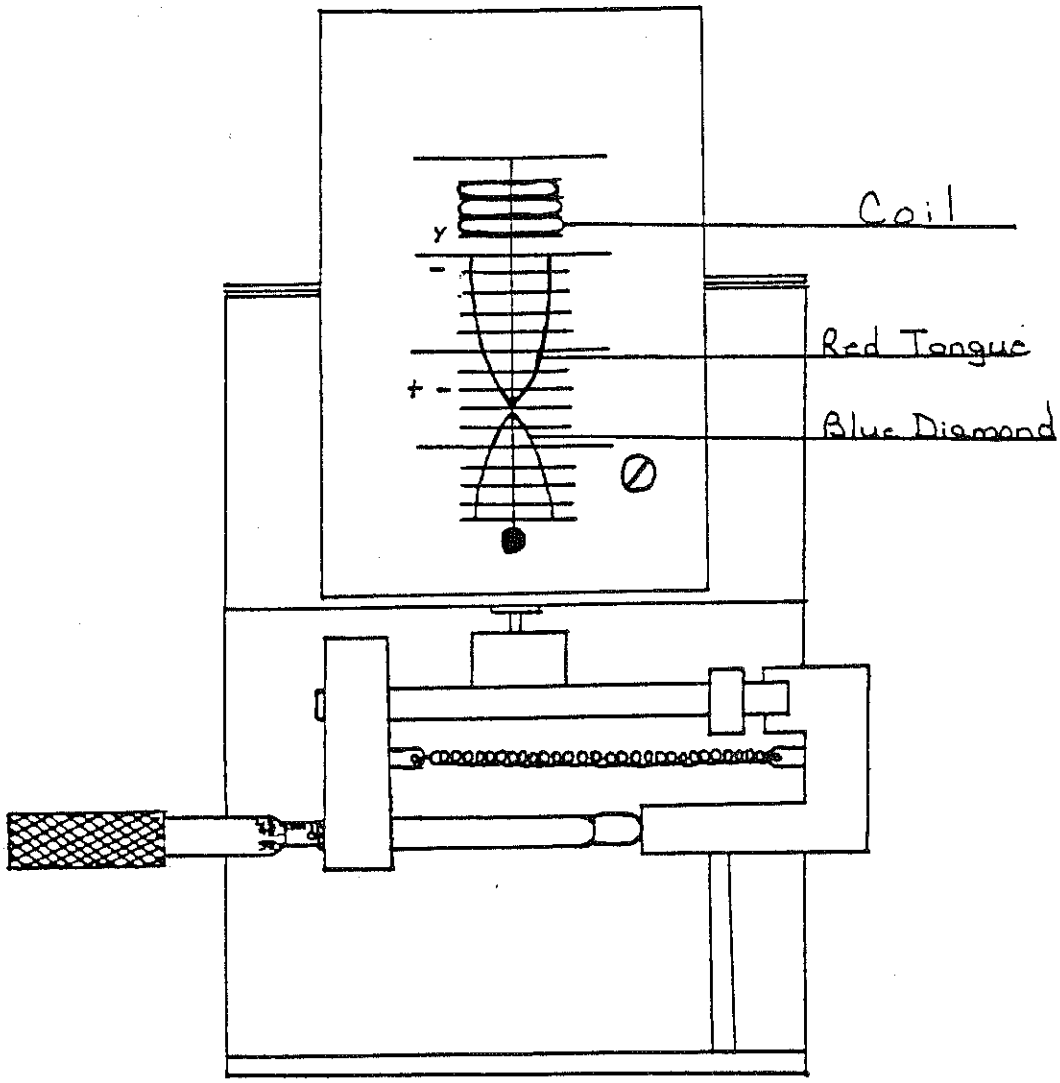
Stock solutions made daily by an analyst using 100x dilution.

APPENDIX 6

CALIBRATION PARAMETERS

Element	Wavelength
Ag	3280.68x2
Al	2373.12x2
As	1936.96x2
Au	2675.92x2
B	2496.78x2
Ba	2335.27x2
Be	3130.42x2
Ca	3158.97x2
Cd	2288.02x3
Co	2286.16x2
Cr	2677.16
Cu	3247.54x2
Ca	3933.66x2
Fe	2714.41
Fe	2599.40x2
K	7664.91
Li	6707.84
Mg	2790.79
Mg	2795.53x2
Mn	2949.20
Mo	2020.30x2
Na	3302.37x2
Ni	2316.04x2
Os	2255.85x2
P	2149.14x3
Pb	2203.53x2
Pt	2036.46x3
Tl	1908.64x3
Ti	3349.41x2
Sb	2068.38x2
Se	1960.26x2
Si	2881.58x2
Sn	1899.90x2
Sr	2152.84x2
V	2924.02x2
Na	5895.92
Zn	2138.56x2
Y	3710.29x2

APPENDIX 7



ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-4
SOP Number: LM-RMA-2006
Title: Metals: GFAA Analysis

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

Dynamic Linear Range:	The dynamic linear range for each analyte is addressed in Attachment 1 of the SOP, page 13 of 15. It is defined by the linear range of standard analyte concentrations.
Section 7.:	Concentrations of the stock standard solutions and working standard solutions will be indicated in the reporting package.
Section 9.1.4:	Duplicate analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.
Section 9.1.5:	Matrix spike analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.
Section 9.2:	SW-846 methods, on which many Enseco methods are based, do not contain control limits for matrix spike and replicate analysis. The SW-846 methods do however contain limits on calibration checks. These checks are ±20 percent or greater, thus indicating that the accuracy limit on a matrix spike can never be greater than 80 to 120 percent. Enseco believes that the control limits indicated in QAPjP Appendix A-11 are realistic, based on the information in the methods and internally developed quality assurance data.

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1. Scope and Application

1.1 Analytes

The four metals most commonly analyzed by Graphite Furnace Atomic Absorption Spectrophotometry (GFAA) are Arsenic, Lead, Selenium, and Thallium.

Other elements that may be analyzed include (but are not limited to) Antimony, Cadmium, Chromium, and Silver.

1.2 Reporting Limits

Reporting limits range from 0.5 to 10 ug/L and ug/kg according to the analyte. Instrument detection limits (IDL) are also available. Consult the specific analyte information at the end of this SOP for details.

Prepared by: David B. Roberts, Jr.	Date: March 3, 1991
Management Approval: David B Roberts Jr	Date: 6-27-91
QA Officer Approval: Willie J Johnson	Date: 06/27/91

1.3 Applicable Matrices

This method is applicable to the analysis of metals in dissolved, recoverable and total water samples, as well as digests of soils, wastes and leachates.

1.4 Dynamic Range

The dynamic range is element specific. For the four most common analytes, it extends to 100 ug/L. Higher concentrations may be analyzed by dilution of the sample. Consult the specific analyte information at the end of this SOP for details.

1.5 Analysis Time

Approximately 20 samples per instrument-shift can be analyzed. It is common practice to run two instruments simultaneously. Sample preparation time is not included.

2. Method Summary

2.1 The sample is first digested according to the appropriate method. Dissolved water samples are analyzed without digestion unless specifically requested by the client.

2.2 An aliquot of the prepared samples is automatically injected into an electrothermally heated graphite furnace which is programmed through a series of drying and charring stages. The element of interest is then atomized by a rapid increase in the temperature of the furnace.

2.3 Radiation from a hollow cathode lamp (HCL) or an electrodeless discharge lamp (EDL) is focused through the graphite furnace. The lamp radiates light at the wavelengths specific to the element being analyzed; atoms of the element in the furnace absorb light at the characteristic wavelength. The absorbance is measured electronically.

2.4 The concentration of the element is calculated by comparing the sample absorbance to the absorbances measured for standards containing known concentrations of the element. This calculation is performed by the instrument.

3. Comments

3.1 Interferences

Any substance which alters the atomization or absorption characteristics of the sample or absorbs light at the same wavelength as the metal being analyzed is a potential interference. Specific interferences are discussed in the individual element procedures.

3.1.1 Chemical and physical interferences are prevalent when analyzing samples using these methods.

3.1.2 Many substances produce non-specific background absorption which must be compensated for by the use of a background correction system, either Deuterium Arc or Zeeman.

3.1.3 Samples are highly subject to contamination at the trace levels of interest in this method. Deionized water and high-purity acids are to be used unless otherwise specified.

3.1.4 Autosampler trays are to be kept covered during analysis.

3.1.5 All work areas used to prepare standards and spike samples are to be cleaned before and after each use.

3.2 Helpful Hints

A summary of the operating conditions used is given in a table at the end of this SOP. For more detail, consult the procedures for the individual elements. Instrument-specific information can be found in the instructions for the instrument being used.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco/RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.

4.2 Wear gloves and apron when handling concentrated acids. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up any spills immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your supervisor or safety officer of any spills or exposures.

4.3 The graphite furnace reaches **EXTREMELY** high temperatures. Be sure that it has cooled sufficiently before removing the tube.

4.4 Radiation emitted by the tube during the atomization step is harmful to the eyes. **DO NOT** look at the tube during atomization.

4.5 The furnace should be properly vented with an exhaust hood directly over the furnace chamber.

4.6 Samples and other solutions containing high concentrations of toxic materials must not be flushed down the sinks, but are to be disposed of in suitable waste containers.

4.7 Refer to the RMAL safety manual for additional precautions and instructions for working in the metals lab.

5. Sample Collection and Preservation

5.1 Dissolved water samples should be filtered at the time of collection, preserved with nitric acid to a pH of less than 2 and stored in clean glass or plastic bottles.

5.2 For collection and preservation information of other matrices, consult the appropriate prep SOPs. See section 8.1.

5.3 The holding time for all metals described by this SOP is 6 months.

6. Apparatus

6.1 Atomic absorption spectrophotometer equipped with graphite furnace, background correction system, autosampler and printer. Examples include:

6.1.1 Perkin Elmer Model 2380 with HGA 400 Graphite Furnace and AS 40 Autosampler.

6.1.2 Perkin Elmer Model 5000 Zeeman with HGA 500 Graphite Furnace and AS 40 Autosampler.

6.1.3 Perkin Elmer 5100 Zeeman with HGA 600 Graphite Furnace and AS 60 Autosampler and Epson computer.

6.2 Electrodeless discharge lamps and power supply, or hollow cathode lamps, specific to the element(s) being analyzed.

6.3 Oxford and Eppendorf pipettors, various sizes, with disposable tips.

6.4 Autosampler cups.

6.5 Graphite tubes, platforms, and other supplies.

6.6 Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

7.1 Standards

7.1.1 Preparation of standard solutions is element specific. Consult the individual element SOPs for details. (See Attachment 1)

7.2 Matrix modifiers

All elements require the addition of a matrix modifier to the sample in order to control interferences. (See Attachment 1 and 3)

7.3 EPA ICV Solution

This solution is used for the Initial Calibration Verification (ICV). It is analyzed immediately following standardization. This solution is obtained from the EPA through EMSL (Las Vegas) by request.

7.4 CCV Solution

This solution is used for Continuing Calibration Verification (CCV). It is analyzed at a frequency of at least 10% (one every 10 samples) during an analytical run. This solution is usually the mid-range standard and prepared daily.

7.5 ICB/CCB Solution

This solution is used for the Initial Calibration Blank (ICB) and the Continuing Calibration Blank (CCB). It is analyzed after the ICV at a frequency of at least 10% (one every 10 samples) during an analytical run. This solution consists of 1% nitric acid plus any matrix modifier used and is prepared daily.

8. Procedure

8.1 Preparation

8.1.1 Total waters, Total Recoverable waters, EP Tox extracts and TCLP extracts are digested using SOP: LM-RMA-2002.

8.1.2 Soils and Wastes are digested using SOP: LM-RMA-2029.

8.2 Instrument Set-Up and Calibration

Instructions vary according to instrument and element. General instructions are given here; consult the appropriate procedures and manuals for the specific analyte being measured and instrument used.

8.2.1 Turn on the main power, cooling water, and argon.

8.2.2 Install the proper lamp and adjust to the proper power setting.

8.2.3 Set the correct wavelength and slit for the element being analyzed.

8.2.4 **CAREFULLY** inspect and clean the quartz windows of the graphite furnace.

8.2.5 After warming up for 10 minutes, optimize the instrument energy throughput.

8.2.6 Make sure that the energy can be turned up past the autogain energy. If problems are encountered achieving the proper energy level, re-optimize the instrument. If this fails to help, then the lamp probably needs to be replaced or the furnace realigned.

8.2.7 Observe the baseline and re-optimize the instrument if it is unstable. If the baseline is still unstable, the lamp probably needs to be replaced.

8.2.8 Turn on the background corrector if using a D2 background correction system. Readjust the lamp power setting, and allow to warm up for 10 minutes.

8.2.9 Observe the baseline again. If it is unstable, re-optimize the instrument. If this fails, check the furnace alignment and readjust if necessary. If the problem persists, turn up the EDL power supply 1 to 2 watts. The deuterium lamp may also need to be replaced.

8.2.10 Inspect the graphite tube and platform, replace if necessary. Clean the contact rings, injection tip and outside of the graphite furnace.

8.2.11 Program the autosampler, furnace, and AA according to the procedures for the instrument being used and the metal being analyzed.

8.2.12 Blanks and calibrations standards must contain any matrix modifier in the same concentration as is in the samples. This is usually added by the instrument using the "Alternate Volume" function or equivalent.

8.2.13 A Calibration Blank and three standards are analyzed.

8.2.2.1 Run blanks successively until the baseline is stable.

8.2.2.2 Calibrate the instrument using the standards indicated for in the procedure for the given instrument and element.

8.2.14 Analyze the EPA ICV, CCV and ICB solutions. If these are not within 10% of the expected value, corrective action must be taken before proceeding.

8.3 Analysis

8.3.1 Add the matrix modifier to all samples

8.3.2 Before analyzing any samples, run the Prep Blank (if applicable) and DCS's. If these are not within acceptable limits, corrective action must be taken before proceeding.

8.3.3 An analytical spike is to be performed on **EACH** sample. See section 9.2 for acceptance criteria.

8.3.4 Samples exceeding the highest standard are to be diluted and reanalyzed or the results taken from the ICP. Dilutions must be made in such a way that the diluted sample contains the same concentration of acid and any matrix modifier as the undiluted sample.

8.3.5 A Blank (CCB) and standard check (CCV) must be performed after every 10 or less samples. If these are not within 10% of the true value, corrective action must be taken and all samples analyzed since the previous "in control" CCV must be reanalyzed.

8.3.6 A final CCB and CCV should be run.

8.4 Conclusion

8.4.1 After completing the analytical run, follow the shut-down procedure for the specific instrument used. This will include removing and storing the lamp, placing the autosampler, graphite furnace and spectrophotometer on stand-by and turning off the gas and water.

8.4.2 Complete **ALL** paperwork neatly and in a timely manner. Data packages should include: a cover sheet with all samples and QC information listed, a review checklist filled out by the analyst, all special instructions, an anomaly form describing the analysis, and all benchsheets with samples and results to be entered into LIMS highlighted and all instrument printouts.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A Prep Blank is required with every batch of 20 or less samples. Dissolved samples will not have a prep blank.

9.1.2 Two DCS samples are required with each batch of 20 or less samples.

9.1.3 A blank (CCB) and standard check (CCV) are required after every 10 or less samples.

9.1.4 Duplicates may be required for project specific QC.

9.1.5 Matrix Spikes may be required for project specific QC.

9.1.6 Analytical spikes are to be performed on **ALL** samples.

9.2 Acceptance Criteria

9.2.1 Method blanks should be less than the reporting limit. If the method blank is more than two times the reporting limit the supervisor should be notified for corrective action.

9.2.2 Average DCS recovery must be 75 - 125%.

9.2.3 The RPD for the DCS must be less than or equal to 20%.

9.2.4 The EPA ICV and all standard checks must be within 10% of the expected value.

9.2.5 There are no acceptance criteria for project specific QC.

9.2.6 Analytical Spike Recovery

(See Flow diagram)

9.2.6.1 Sample reads **below** the reporting limit (RL)

Recovery	Action
< 40%	Dilute, reanalyze
>=40% and <80%	Report ND at 2x RL
>=80% and <=120%	Report ND at RL
Recovery >120	Report ND at 2x RL

9.2.6.2 Sample reads **at or above** the reporting limit (RL)

Recovery	Action
< 40%	Dilute, reanalyze
>=40% and <80%	Dilute, reanalyze
>=80% and <=120%	Report result at RL
> 120%	Dilute, reanalyze

NOTE: MSA = Method of standard addition - i.e. be applied under supervisor discretion.

(See Attachment 2)

9.2.7 Dilutions

When a sample must be diluted the dilution chosen will be determined by the analyst's best judgement and should place the analyte concentration in the optimum range of the calibration curve. The sample should not be over-diluted.

9.3 Corrective Action Required

9.3.1 Corrective action taken when the acceptance criteria are not met may include: reanalysis of samples not bracketed by acceptable QC, restandardization of the instrument and reanalysis, or repreparation of the samples.

9.3.2 Follow the corrective actions outlined in the current Enseco QAPP Manual.

10. Calculations

10.1 The instrument automatically calculates sample concentrations from the absorbances.

10.2 Multiply the concentration from the instrument by any dilution factors made during sample preparation and analysis.

10.3 To convert from ug/L (ppb) to mg/L, divide by 1000.

11. Reporting Requirements

11.1 Units

Reporting units are mg/L for aqueous samples and mg/kg for non-aqueous samples. As a convenience, units of ug/L (ppb) may used during analysis.

11.2 Limits

Samples less than the reporting limit are reported as N.D.

11.3 Significant Figures

Element	Rep. Limit	0.0001-0.0009	0.001-0.009	0.01-0.09	> 0.10
Arsenic	0.005 mg/L	--	1	2	2
Lead	0.005 mg/L	--	1	2	2
Selenium	0.005 mg/L	--	1	2	2
Thallium	0.005 mg/L	--	1	2	2
Antimony	0.01 mg/L	--	--	1	2
Cadmium	0.0005 mg/L	1	2	2	2
Chromium	0.005 mg/L	--	1	2	2
Silver	0.0005 mg/L	1	2	2	2

11.4 LIMS Data Entry

The usual standards for data entry apply.

11.5 Anomalies

11.5.1 Samples that contain high levels of soluble salts may form crystals upon drying in the furnace and require dilution to obtain acceptable spike recoveries.

11.5.2 High concentrations of non-target compounds may cause poor spike recovery. These samples must be diluted until the spike recovery is acceptable and the reporting limit raised accordingly.

11.5.3 Samples containing significant concentrations of organic material may not pipette smoothly into the furnace or be deposited on the platform properly. These samples must also be diluted or digested before analysis.

11.5.4 Samples requiring large dilutions due to the presence of high concentrations of analyte may be reported using alternate techniques, ie. Flame AA or ICP.

12. Review Requirements

12.1 All data packages must be complete, signed and dated by both the analyst and the peer reviewer. Both the coversheet and the benchsheet should state the analyst's name, the date and type of analysis performed and the instrument used.

12.2 Benchsheets must contain all calibration information, true values for all QC solutions and recoveries for all QC and analytical spikes.

12.3 Verify that calculations were done correctly by randomly checking a few of them.

13. References

13.1 Source Method: USEPA SW-846, "Test Methods for Evaluating Solid Waste", Chapter 3, 7000-series methods, 3rd Edition, 1986.

13.2 Related Documents

13.2.1 Methods for Chemical Analysis of Water and Wastes, EPA 600 Methods, revised March 1983.

13.2.2 Instruction manuals for Perkin Elmer Model 2380, Zeeman 5000 and Zeeman 5100

13.3 Deviations from Source Method and Rationale

Consult individual element procedures.

ATTACHMENT 1

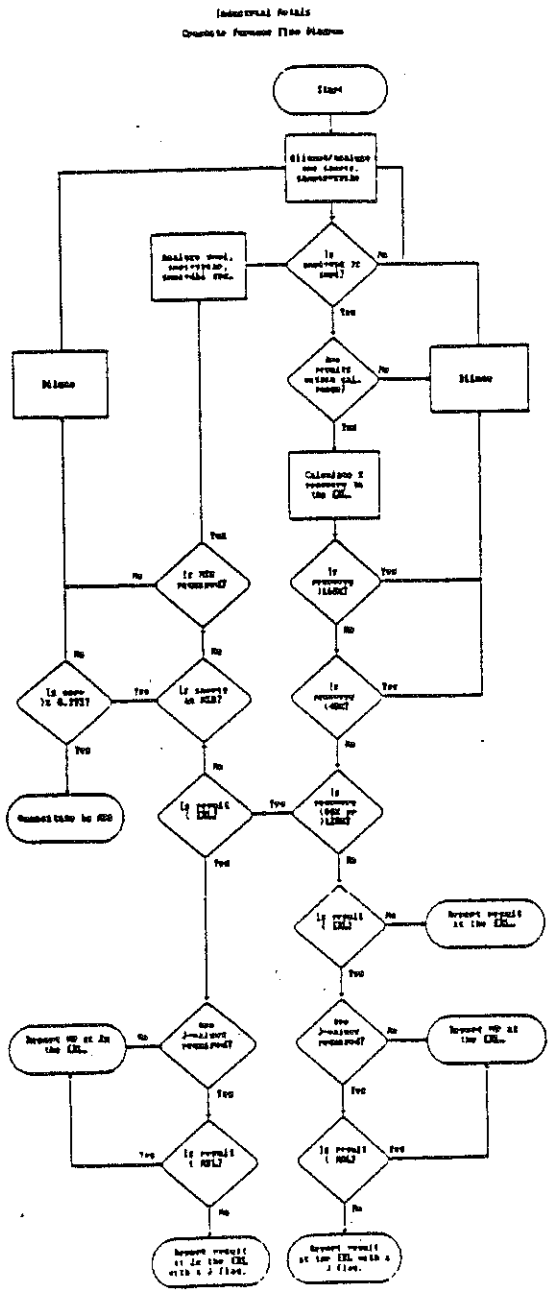
STANDARD OPERATING CONDITIONS FOR METALS
(GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROPHOTOMETRY)

Element	EPA Ref.	SOP Ref.	Wavelength	Slit	Matrix Modifier
Arsenic	7060	LM-RMA-2007	193.7 nm	0.7	$Ni(NO_3)_2$
Lead	7421	LM-RMA-2013	283.3 nm	0.7	$(NH_4)_2HPO_4$
Selenium	7740	LM-RMA-2019	196.0 nm	0.7	$Ni(NO_3)_2$
Thallium	7841	LM-RMA-2026	276.8 nm	0.7	H_2SO_4
Antimony	7041	LM-RMA-2005	217.6 nm	0.2	$Ni(NO_3)_2$
Cadmium	7131	LM-RMA-2011	228.8 nm	0.7	$(NH_4)_2HPO_4$
Chromium	7191	LM-RMA-2036	357.9 nm	0.7	$Mg(NO_3)_2$
Silver	7761	LM-RMA-2023	328.1 nm	0.7	PO_3

Element	Rep. Limit	Standard Concentrations	Analytical Spike(ug/L)
Arsenic	0.005 mg/L	0, 20, 50, 100	20
Lead	0.005 mg/L	0, 20, 50, 100	20
Selenium	0.005 mg/L	0, 20, 50, 100	20
Thallium	0.005 mg/L	0, 20, 50, 100	20
Antimony	0.01 mg/L	0, 20, 50, 100	20
Cadmium	0.0005 mg/L	0, 1, 2.5, 5	1
Chromium	0.005 mg/L	0, 20, 50, 100	20
Silver	0.0005 mg/L	0, 1, 2.5, 5	1

SOP No.:	Revision No.:	Effective Date:
LM-RMA-2006	1.0	March 3, 1991

ATTACHMENT 2



ATTACHMENT 3

MATRIX MODIFIERS
MARCH 1991

Element	SW846	RMAL	Concentrations
Sb Z	(7041) 0.1% $NiNO_3$	0.1% Ni	20 uL of 7.5% $NiNO_3$ to 1.5 mL sample (cup)
As Z	(7060) 0.1% $NiNO_3$	0.1% Ni	20 uL of 7.5% $NiNO_3$ to 1.5 mL sample (cup)
Cd D2	(7131) ?x40% $(NH_4)_2$ HPO_4	1% PO_4	20 uL of 75% $(NH_4)_2$ to 1.5 mL sample (cup)
Cr D2	(7191) ?x1.18% $Ca(NO_3)_2$ 4 H_2O	0.025% Mg $(NO_3)_2$	20 uL of 1.87% $Mg(NO_3)_2$ in 1.5 mL sample (cup)
Pb D2	(7421) 0.85% HPO_4	1% PO_4	20 uL of 75% $(NH_4)_2$ HPO_4 to 1.5 mL sample (cup)
Ag D2	(7761) Uses a different prep	1% PO_4	20 uL of 75% $(NH_4)_2$ HPO_4 to 1.5 mL sample (cup)
Se Z	(7740) 0.1% Ni	0.1% Ni and 0.125% $Mg(NO_3)_2$	20 uL of 7.5% $NiNO_3$ and 9.4% $Mg(NO_3)_2$ to 1.5 mL sample (cup)
Tl D2	(7841) ?x0.025% $PdCl_2$ and ?x1% HNO_3	0.01% H_2SO_4	20 uL of 0.75% H_2SO_4 to 1.5 mL sample (cup)

Subject or Title: Total Metals: Digestion Preparation (Soils/Wastes)

SOP No.: LM-RMA-2029

Revision No.: 1.01

Effective Date: January 31, 1991

Supersedes: LM-RMA-2029, Revision 1.0

1. Scope and Application

1.1 Analytes

This method is an acid digestion procedure used to prepare a soil or waste sample for analysis by flame or Graphite Furnace Atomic Absorption Spectroscopy (AAS) or by Inductively Coupled Plasma (ICP) spectroscopy for a variety of trace metals. Mercury is not included.

1.2 Reporting Limits

Refer to the individual analysis SOPs.

1.3 Applicable Matrices

This method is applicable to soil and industrial waste matrices.

1.4 Dynamic Range

Refer to the individual analysis SOPs.

1.5 Analysis Time

The preparation time for one batch of 20 samples and required QC is approximately 8 hours. Refer to the individual analysis SOPs for approximate analysis times.

Prepared by: Sherman Gray/Will Pratt	Date: January 31, 1991
Management Approval: Will W Pratt II	Date: 2/4/91
QA Officer Approval: Linda Sign	Date: 1/31/91

2. Method Summary

2.1 A representative 1 g sample aliquot is weighed into a 250 mL beaker. Dilute nitric acid is added, the beaker covered with a watch glass and the sample heated to 95°C.

2.2 The sample is cooled and digested with dilute hydrogen peroxide. For GFAA analysis, no futher digestion is performed.

2.3 For FLAA or ICP analysis, the sample is cooled and digested with dilute hydrochloric acid.

2.4 All samples are cooled, filtered and diluted to a final volume of 100 mL.

3. Comments

3.1 Interferences

3.1.1 The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfering, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

3.1.2 Failure to homogenize the sample well prior to removal of the desired aliquot may yield erroneous results.

3.2 Helpful Hints

3.2.1 The terms "flame" and "furnace" are used frequently in this SOP. Pay particular attention to such terminology to avoid misinterpretation.

3.2.2 Samples requiring a "dry weight" analysis or calculation (as specified in special instructions) are prepared using the wet weight equivalent. See section 10.1.

3.2.3 Bring samples up to volume (mass) within 1% of the desired mass. For example, bring a soil digest to within 1 mL of the desired final volume (mass) of 100 mL.

4. Safety Issues

4.1 Proper protective gear (i.e., safety glasses, face shield, gloves, and rubber apron) should be worn when using acid or peroxide.

4.2 All sample transfers and digestions should take place in a properly vented hood, and spills should be cleaned up immediately.

4.3 Nitric acid, hydrochloric acid and hydrogen peroxide are corrosive and will cause skin irritation. Avoid contact with eyes, skin and clothing. Wash thoroughly with copious amounts of water.

4.4 All waste generated from the test will be placed into an approved, correctly labelled waste container.

4.5 The RMAL safety manual should be referred to for more detailed cautions and instructions.

5. Sample Collection, Preservation and Holding Times

5.1 Samples should be collected in glass or plastic containers.

5.2 Samples should be refrigerated at $4^{o}C$ until the time of preparation.

5.3 The holding time for all metals analyzed by atomic spectroscopy is 180 days (6 months), except mercury. The preparation and analysis for mercury is described in SOP LM-RMA-2005.

6. Apparatus

6.1 Hot plate, capable of 100° C.

6.2 Miscellaneous pipettes.

6.3 Miscellaneous benchsheets.

6.4 Calculator.

6.5 250 mL glass beakers with watch glasses.

6.6 Top-loading balance, 0.01 g capability.

6.7 Miscellaneous filter apparatus.

6.8 Plastic bottles (1% HNO_3 rinsed).

7. Reagents and Standards

7.1 ASTM Type II water (Milli Q or equivalent).

7.2 Nitric acid, (HNO_3), concentrated, trace metal grade.

7.3 Nitric acid, (HNO_3), 75% solution (v/v): Carefully add 1500 mL concentrated nitric acid to 500 mL water.

7.4 Nitric acid, (HNO_3), 1% solution (v/v): Carefully add 20 mL concentrated nitric acid to 1980 mL water.

7.5 Hydrochloric acid, (HCl), concentrated, trace metal grade.

7.6 Hydrochloric acid, (HCl), 50% solution (v/v): Carefully add 1000 mL concentrated hydrochloric acid to 1000 mL water.

7.7 Hydrogen peroxide, (H_2O_2), reagent grade.

7.8 Duplicate control standard solutions (DCS).

7.8.1 Samples prepared for analysis by Graphite Furnace AAS use the GFAA duplicate control standard solutions "A" and "B".

7.8.2 Samples prepared for analysis by Inductively Coupled Plasma use the ICP duplicate control standard solutions "A", "B", "C" and "D".

7.8.3 Samples prepared for analysis by Flame AAS use the ICP duplicate control standard solutions "A", "B", "C", "D" and "E".

8. Procedure

8.1 Instrument Set-up and Calibration

8.1.1 Turn on hot plate, set for 95°C (see SOP: LP-RMQ-0053 for calibration check procedure). Allow 1/2 hour to preheat.

8.1.2 Verify the calibration of the balance and pipette.

8.2 Preparation

8.2.1 Identify the test and samples to be digested using the backlogs.

8.2.2 Check the special instructions for each sample and print a copy if they apply.

NOTE: Be aware of requests for Dry Weight (prep, analysis, calculation, etc.). Consult your supervisor.

8.2.3 Locate and sign out the samples in the walk-in cooler. If a sample cannot be located, consult your supervisor.

8.2.4 Assign a QC Lot Number and fill out the appropriate bench sheet (separate bench sheets are completed for GFAA and ICP/FLAA digestions).

8.2.5 Update the backlog to reflect which samples are in progress.

8.2.6 Clearly label each beaker as appropriate (red stickers are used for GFAA preps and green stickers are used for ICP/FLAA preps).

8.3 Analysis

8.3.1 Homogenize the sample as much as possible with a clean plastic spoon or spatula and weigh a 1.0 g portion into a labelled 250 mL beaker. Report sample specific anomalies and/or comments on the bench sheet.

8.3.2 Carefully add 20 mL of 75% HNO_3 to the sample and cover the beaker with a watch glass. Heat on a hot plate to 95° and allow to reflux and evaporate to 10 mL volume (approximately 1-2 hours). Remove and cool.

NOTE: DO NOT let the sample boil or the volume reduce to less than 5 mL.

8.3.3 Add 5 mL of 30% H_2O_2, return to the hot plate and heat for 30 minutes. Remove and cool. Repeat once. Continue heating the sample until the volume is reduced to approximately 10 mL.

NOTE: DO NOT let the sample boil or the volume reduce to less than 5 mL.

8.3.4 For Graphite Furnace AAS analysis, add 10 mL Milli Q water and heat the sample for 15 minutes. Remove and cool.

8.3.5 For Flame AAS or ICP analysis, add 10 mL 50% HCl and heat the sample for 30 minutes. Remove and cool.

8.3.6 Label plastic bottles using available computer program.

8.3.7 Weigh each sample digest bottle and record that weight under the bottle label. Rinse the bottle with 1% HNO_3.

8.3.8 Filter the sample through filter paper (Whatman 41 or equivalent that has been pre-rinsed with 1% HNO_3) into the pre-weighed, acid-rinsed poly bottle.

8.3.9 Rinse the beaker, watch glass and the filter paper sparingly, but well, with Milli Q water to ensure complete sample transfer. See your supervisor for specific technique.

8.3.10 Adjust the sample volume to a final mass of 100 g with Milli Q water.

8.3.11 Check that the bottle cap and bottle are labelled identically and cap the bottle. The sample is now ready for analysis.

8.4 Conclusion

8.4.1 Complete all paperwork and release prep from "logged" to "done" in LIMS. Consult your supervisor.

8.4.2 Place samples in properly labelled box (see attached example) and deliver to appropriate analysis shelf.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank shall be prepared for each 20 samples digested or for each batch, whichever is more frequent. The blank is prepared by taking 1.0 g of Milli Q water and digesting it as described in section 8.3.2 through 8.3.11.

9.1.2 Duplicate control samples (DCS) shall be prepared for each 20 samples digested or for each batch, whichever is more frequent. These are prepared by spiking 1.00 mL of each of the DCS stock solutions into 1.0 g of Milli Q water and digesting them as described in section 8.3.2 through 8.3.11.

NOTE: There are two separate DCS solutions for Graphite Furnace AAS, four separate DCS solutions for ICP, and five separate DCS solutions for Flame AAS digests. Each DCS solution has its own verification number. See section 7.8.

9.1.3 Client specific sample duplicates (DU), matrix spikes (MS) and matrix spike duplicates (SD) will be prepared only upon client request and will then appear on the prep scheduling backlog sheet.

9.1.4 See SOP: M-EQA-0002 and the Enseco QAPP.

9.2 Acceptance Criteria

Refer to the appropriate RMAL individual analysis SOP for each specific analyte.

9.3 Corrective Action Required

Follow the corrective actions outlined in the current Enseco QAPP manual or consult your supervisor.

10. Calculations

10.1 The mass of wet sample to be prepped for "dry weight" reporting is calculated as:

$$Mw = \frac{1.0\ g}{(TS/100)}$$

Where Mw = sample mass wet and
TS = percent total solids and
assuming the desired dry mass
is 1.0 grams.

For example, a sample is 50% total solids and a "dry weight" prep is required:

$$Mw = \frac{1.0\ g}{(50/100)} = 2.0\ g\ (wet)$$

10.2 The final dilution factor is calculated as follows:

DF = Dilution Factor
M = Initial sample mass
V = Final volume of digest

$$DF = \frac{V}{M}$$

NOTE: On a "dry weight" prep, the "wet weight" equlivalent of 1.0 g dry has no impact on the initial sample weight used in the calculation of the final dilution factor.

11. Reporting Requirements

11.1 All data packets will be compiled in the following order: Bench sheet, total solids bench sheet copy (when applicable) and a copy of the special instructions.

11.2 The prep analyst name, prep date, QC lot number, sample number, acknowledgment of special instructions, anomalies observed, dry weight equivalent mass, sample mass, final volume, dilution factor, spike solution verification numbers and comments are recorded on the prep bench sheet (see attached example).

11.3 Calculate and report all "dry weight" equivalents on the bench sheet (when applicable).

12. Review Requirements

12.1 Verify special instructions were followed.

12.2 Verify dilution factors were correctly calculated (including "dry weight" preps, when applicable).

13. References

13.1 Source Method: Method 3050, SW-846 - Test Methods for Evaluating Solid Waste, 3rd Edition, 1986.

13.2 Related Documents

Method 200.7. Inductively Coupled Plasma - Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes. "Methods of Chemical Analysis of Water and Wastes (MCAWW)", EPA-600/4-79-020, March, 1983.

13.3 Deviations from Source Method and Rationale

13.3.1 For convenience sake, (step 8.3.2 of this document) add 20 mL of 75% HNO_3 and heat for 1 to 2 hours in lieu of the following:

Add 10 mL 1:1 HNO_3, reflux 15 minutes, add 5 mL concentrated HNO3 and reflux for 30 minutes.

13.3.2 For convenience sake, (step 8.3.3 of this document) add 5 mL of 30% H_2O_2 and heat for 30 minutes, then repeat once in lieu of the following:

Add 2 mL of H_2O and 3 mL H_2O_2, heat until effervescence subsides, continue to add 30% H_2O_2 in 1 mL aliquots until effervescence is minimal.

13.4 Updates to SOP (Original to Revision 1.01)

13.4.1 Addition and strength of nitric acid.

13.4.2 Addition of hydrogen peroxide.

13.4.3 All other changes simplify working details of the SOP.

13.4.4 Addition of hot plate calibration SOP.

Enseco

STANDARD
OPERATING
PROCEDURE

Subject or Title: Total Recoverable, Dissolved and Total Metals: Acid Digestion (Aqueous samples/EP Tox. extracts)

SOP No.:	Revision No.:	Effective Date:
LM-RMA-2002	2.0	January 22, 1990

Supersedes:
Revision 1.0 (February 13, 1989)

1. Scope and Application

1.1 Analytes

These acid digestion methods are used to prepare aqueous samples and Extraction Procedure (EP) extracts for analysis of a variety of trace metals by Graphite Furnace Atomic Absorption spectroscopy (GFAA), Inductively Coupled Argon Plasma spectroscopy (ICP) or Flame Atomic Absorption spectroscopy (FLAA). Mercury is not included. Refer to the individual analysis SOPs for specific analytes.

1.2 Reporting Limits

Refer to the individual analysis SOPs.

1.3 Applicable Matrices

These methods are applicable to surface and ground water samples, industrial and domestic waste samples and EP extract solutions.

1.4 Dynamic Range

Refer to the individual analysis SOPs.

1.5 Analysis Time

The approximate preparation time for one batch of 20 samples and required QC is approximately 7 hours for any of the methods. Refer to the individual analysis SOPs for approximate analysis times.

Prepared by: Sherman Gray/Will Pratt Sherman T Gray	Date: January 19, 1990
Management Approval:	Date: 1/19/90
QA Officer Approval:	Date: 1/19/90

2. Method Summary

2.1 A representative 100 mL aliquot of sample is placed into a 250 mL beaker. Various mineral acids are added and the sample is heated to 95°C to effect dissolution:

Total/EP extract. - GFAA: Nitric acid only.

Total/EP extract. - ICP/FLAA: Nitric and hydrochloric acids.

Dissolved/Tot. Recov. - ICP/FLAA: Nitric acid only.

NOTE: The standard Enseco procedure for analyzing Dissolved metals is NOT to digest them unless specifically requested by the client or for other valid reasons as determined by management; as a general rule, if a sample being analyzed for Dissolved metals is found to contain sediment or precipitate, the sample will be filtered, analyzed and the results anomalized.

2.2 All samples are cooled, filtered and diluted to a final volume of 100 mL. Anomalies are recorded on the benchsheet and transferred with the samples to the analysis area.

3. Comments

3.1 Interferences

3.1.1 The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper reagent transfering, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

3.1.2 Failure to homogenize the sample well prior to removal of the desired aliquot may yield erroneous results.

3.1.3 Allowing samples to boil or go dry during digestion may result in the loss of volatile metal compounds.

3.2 Helpful Hints

3.2.1 It is only required to bring samples to within 1% of the desired volume. For example, bring a sample digest to within 1 mL of the desired final volume of 100 mL.

3.2.2 Teflon boiling chips should be used when digesting samples that contain sediment to help prevent them from bumping.

4. Safety Issues

4.1 Proper protective gear (i.e., safety glasses, face shield, gloves, and rubber apron) should be worn when using acids.

4.2 All sample transfers and digestions should take place in a properly vented hood, and spills should be cleaned up imediately.

4.3 Nitric acid and hydrochloric acid are corrosive and will cause skin irritation. Avoid contact with eyes, skin and clothing. Wash thoroughly with copious amounts of water.

4.4 All waste generated from the test will be placed into an approved waste container.

4.5 The RMAL safety manual should be referred to for detailed cautions and instructions.

5. Sample Collection, Preservation and Holding Times

5.1 Samples should be collected in prewashed glass or plastic containers.

5.2 Total Recoverable and Total Metals - Samples should be acidified with nitric acid to pH < 2 at the time of collection.

5.3 Dissolved Metals - Samples should be filtered through 0.5 micron filter and then acidified with nitric acid to pH < 2 at the time of collection.

5.4 The holding time for all metals analyzed by atomic spectroscopy is 180 days (6 months), except mercury. The preparation and analysis for mercury is described in SOP LM-RMA-2005.

6. Apparatus

6.1 Hot plate, capable of 100°C.

6.2 Miscellaneous pipettes.

6.3 Miscellaneous benchsheets.

6.4 Calculator.

6.5 250 mL glass beakers with watch glass covers.

6.6 Top-loading balance, 0.01 g capability.

6.7 Miscellaneous filter apparatus.

6.8 Plastic bottles (1% HNO_3 rinsed).

7. **Reagents and Standards**

7.1 ASTM Type II water, (Milli Q or equivalent).

7.2 Nitric acid, (HNO_3), concentrated, trace metal grade.

7.3 1% Nitric acid solution: Add 20 mL concentrated nitric acid to 2000 mL Milli Q water. Cap, mix well.

7.4 Hydrochloric acid, (HCl), concentrated, trace metal grade.

7.5 Duplicate Control Standard stock solutions (DCS):

7.5.1 Samples prepared for analysis by Graphite Furnace AAS use the GFAA duplicate control standard solutions "A" and "B".

7.5.2 Samples prepared for analysis by Inductively Coupled Plasma use the ICP duplicate control standard solutions "A", "B", "C" and "D".

7.5.3 Samples prepared for analysis by Flame AAS use the ICP duplicate control standard solutions "A", "B", "C", "D" and "E". Consult your supervisor.

8. **Procedure**

8.1 Instrument Set-up and Calibration

8.1.1 Turn on hot plate, set for 95°C (see SOP: LP-RMA-0053 for calibration check procedure). Allow 1/2 hour to preheat.

8.1.2 Verify the calibration of the balance and pipettes.

8.2 General Preparation

8.2.1 Identify the test and samples to be digested using the backlogs.

8.2.2 Check the special instructions for each sample and print a copy if they apply.

8.2.3 Locate and sign out the samples in the walk-in cooler. If a sample cannot be located, consult your supervisor.

8.2.4 Assign a QC Lot Number and fill out the appropriate bench sheet (separate bench sheets are completed for GFAA and ICP/FLAA digestions).

8.2.5 Update the backlog to reflect which samples are in progress.

8.2.6 Clearly label each beaker as appropriate (red stickers are used for GFAA preps and green stickers are used for ICP/FLAA preps).

8.2.7 Place a labelled 250 mL beaker on a top-loading balance and tare the balance.

8.2.8 Transfer a 100 +/- 1 mL (g) aliquot of homogenized sample into the beaker. Record any anomalies or comments on the benchsheet (see attached example).

8.2.9 Proceed to the appropriate section for the desired method:

Method	Section
Total/EP Tox. - GFAA	8.3
Total/EP Tox. - ICP/FLAA	8.4
Tot. Recov./Dissolved - ICP/FLAA	8.5

8.3 **Digestion: Total and EP Toxicity Metals for GFAA**

8.3.1 Carefully add 5 mL of concentrated HNO_3 to the sample/extract and cover the beaker with a watch glass. Place on hot plate.

8.3.2 Allow the sample to reflux and evaporate to low volume (15-20 mL) while swirling occasionally. NOTE: DO NOT let the sample boil or go dry. Doing so will result in loss of analytes and the sample must be reprepped.

8.3.3 Remove, cool and carefully add another 5 mL of concentrated HNO_3 to the sample.

8.3.4 Return beaker to the hot plate and allow the sample to reflux and evaporate for 30 minutes. NOTE: DO NOT let the sample boil or go dry.

8.3.5 Proceed to section 8.6.

8.4 **Digestion: Total and EP Toxicity Metals for ICP/FLAA**

8.4.1 Carefully add 5 mL of concentrated HNO_3 to the sample/extract and cover the beaker with a watch glass. Place on hot plate.

8.4.2 Allow the sample to reflux and evaporate to low volume (15-20 mL) while swirling occasionally. NOTE: DO NOT let the sample boil or go dry. Doing so will result in loss of analytes and the sample will have to be reprepped.

8.4.3 Remove, cool and carefully add another 5 mL of concentrated HNO_3 to the sample.

8.4.4 Return beaker to the hot plate and allow the sample to reflux and evaporate for 30 minutes. NOTE: DO NOT let the sample boil or go dry.

8.4.5 Remove, cool and carefully add 5 mL of concentrated HCl to the sample.

8.4.6 Return beaker to the hot plate and allow the sample to reflux and evaporate for 15 minutes. NOTE: DO NOT let the sample boil or go dry.

8.4.7 Proceed to section 8.6.

8.5 **Digestion: Total Recoverable and Dissolved Metals for ICP/FLAA**

NOTE: The standard Enseco procedure for analyzing Dissolved metals is NOT to digest them unless specifically requested by the client or for other valid reasons as determined by management; as a general rule, if a sample being analyzed for Dissolved metals is found to contain sediment or precipitate, the sample will be filtered, analyzed and the results anomalized.

8.5.1 Carefully add 2 mL of concentrated HNO_3 and 5 mL concentrated HCl to the sample, cover the beaker with a watch glass and place on hot plate.

8.5.2 Allow the sample to reflux and evaporate to low volume (15-20 mL) while swirling occasionally. NOTE: DO NOT let the sample boil or go dry. Doing so will result in loss of analytes and the sample must be reprepped.

8.6 Conclusion

8.6.1 Remove and carefully add 10 mL Milli Q water, swirl to mix and allow to cool.

8.6.2 Label plastic bottles using the available computer program.

8.6.3 Weigh each sample digest bottle and record that weight under the bottle label. Rinse the bottle with 1% HNO_3.

8.6.4 Filter the cooled sample through filter paper (Whatman 41 or equivalent that has been pre-rinsed with 1% HNO_3) into the pre-weighed, acid-rinsed poly bottle.

8.6.5 Rinse the beaker, watch glass and the filter paper sparingly, but well with Milli Q water to ensure complete sample transfer. See your supervisor for specific technique.

8.6.6 Adjust the sample volume (mass) to 100 mL (g) with Milli Q water.

8.6.7 Confirm that the bottle cap and bottle are labelled identically; cap the bottle. The sample is now ready for analysis.

8.6.8 Complete all paperwork and submit to supervisor for immediate approval. Release prep test from "Logged" to "Done" in LIMS.

8.6.9 Place samples in a properly labelled box (see attached example) and deliver to appropriate analysis shelf with all "approved" paperwork.

8.6.10 Clean all glassware and work areas thoroughly.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank shall be prepared for each 20 samples digested or for each batch, whichever is more frequent. The blank is prepared by taking 100 mL (g) of Milli Q water and digesting it as described in the appropriate section.

9.1.2 Duplicate Control Samples (DCS) shall be prepared for each 20 samples digested or for each batch, whichever is more frequent. These are prepared by spiking 1.0 mL of each of the DCS stock solutions into 100 mL (g) of Milli Q water and digesting them as described in the appropriate section.

NOTE: There are two separate DCS solutions for Graphite Furnace AA, four separate DCS solutions for ICP, and five separate DCS solutions for Flame AA digests. Each DCS solution has its own verification number. See section 7.4.

9.1.3 Client specific sample duplicates (DU), matrix spikes (MS) and matrix spike duplicates (SD) will be prepared only upon client request and will then appear on the prep scheduling backlog sheet.

9.1.4 See SOP: M-EQA-0002 and the Enseco QAPP.

9.2 Acceptance Criteria

Refer to the appropriate RMAL individual analysis SOP for each specific analyte.

9.3 Corrective Action Required

9.3.1 Sample digests which have boiled or gone dry must be reprepped due to the potential loss of volatile metal compounds.

9.3.2 Follow the corrective actions outlined in the current Enseco QAPP manual or consult your supervisor.

10. Calculations

The final dilution factor is calculated as follows:

$$DF = \frac{V_f}{V_i}$$

where DF is the dilution factor, V_f is the final digest volume and V_i is the initial sample volume.

11. Reporting Requirements

11.1 Data packages will contain the bench sheet and a copy of the special instructions (where applicable).

11.2 The test, analyst name, prep date, QC lot number, sample number, acknowledgment of special instructions, anomalies observed, sample mass, final volume, dilution factor, spike solution verification numbers and comments are recorded on the prep bench sheet (see attached example).

12. Review Requirements

NOTE: It is the responsibility of the analyst to perform an initial review of the prep information as a part of the analysis so as to alert him to potential problems or difficulties.

12.1 Verify special instructions were followed.

12.2 Verify dilution factors were correctly calculated.

12.3 Verify that anomalies were correctly noted.

13. References

13.1 Source Methods: Method 3005, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy"; Method 3010, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy"; and Method 3020, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy", SW-846 - Test Methods for Evaluating Solid Waste, 3rd edition, 1986.

13.2 Related Documents

13.2.1 Method 200.7, "Inductively Coupled Plasma - Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes", Methods of Chemical Analysis of Water and Wastes (MCAWW), EPA-600/4-79-020, March, 1983.

13.2.2 GFAA Metals analysis SOP: LM-RMA-2006.

13.2.3 ICP Metals analysis SOP: LM-RMA-2037.

13.2.4 FLAA Metals analysis SOP: Currently unavailable.

13.2.5 Enseco QAPP, most recent revision.

13.2.6 Enseco SOP: M-EQA-0002.

13.3 Deviations from Source Methods and Rationale

13.3.1 Methods 3010 and 3020 specify multiple 3 mL additions of HNO_3 until the digestion is "complete". The addition of two (2) 5 mL portions of HNO_3 has been shown to provide equally effective digestive action and will continue to be used for convenience sake.

13.3.2 The analysis of replicate samples (duplicates or "dupes") is recommended at a frequency of 20% depending on the sample load. Due to the fact the acceptance criteria have not been established or recommended, the preparation and analysis of replicate samples is not practiced, with the exception of DCSs (section 9.1.2).

13.4 Updates to SOP (Revision 1.0 to Revision 2.0)

Revision 2.0 primarily reflects the changes brought about by SW-846, 3rd Edition.

13.4.1 The use of hydrogen peroxide as a digestion reagent has been eliminated.

13.4.2 The preparation of samples for Total Recoverable Metals analysis by Graphite Furnace AA (GFAA) has been eliminated.

13.4.3 Format changes including the addition of the Reporting and Review Requirements sections were effected.

INDUSTRIAL INORGANIC CHEMISTRY PREP COVER SHEET

P-M-ICP-AT/AR

Analyst SGRAY Date 19 JAN 90 Lot A Approved [illegible] Date 1/19/90

Sample Number	Special Instructions? Y	N/A	N	Anomalies Observed (legend below)	Sample Mass (g) (A)	Final Volume (mL) (B)	Dilution Factor (B/A)
Blank	---	---	---	----------	100.0 mL	100 mL	1 X
DCS 1	---	---	---	----------	100.0 mL	100 mL	1 X
DCS 2	---	---	---	----------	100.0 mL	100 mL	1 X
ABCD-Ø1			✓		100.0	100.0	1X
-Ø2			✓		↓	↓	↓
-Ø3			✓	5	↓	↓	↓
BCDE-Ø1	✓			1	50.0	50.0	↓
-Ø2	✓			1	50.0	50.0	↓

Spike Solution Verification numbers: (A). ZYXW (B). YXWV C). XWVU (D). WVUT

Comments: Dropped AT 50 mls PER SPECIAL INSTRUCTIONS,

ANOMALY LEGEND

(1). Limited sample volume.
(2). Inhomogenous sample.
(3). Multiphase sample.
(4). Slow to filter after digest.
(5). Layer of sediment present on the bottom of bottle.
(6). Sample aliquot changed color on addition of acids.
(7). Broken/leaking sample bottle before preparation.
(8). The sample was digested using a waste digest method.
(09). Oil layer on top of sample.
(10). Boiling stones used.
(11). Sample is opaque or colored.
(12). Suspended material present.

Q C LOT NUMBER 19-JAN 90 A

P-M-ICP-AT

Project(s)	Sample numbers
ABCD	Ø1, Ø2, Ø3
BCDE	Ø1, Ø2

Prep performed by SGRAY Date 19-JAN 90

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-5
SOP Number: LM-RMA-2005
Title: Mecury Preparation and Analysis

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

A new SOP version is presented with this QAPjP submittal (Revision 1).

Dynamic Range: Dynamic range is 0.0002 ppm to .002 ppm Hg concentration.

Section 7.: Preparation of laboratory control sample and calibration stock solutions are addressed in SOP LS-RMA-4501, now a part of QAPjP Appendix B-5.

Five levels of standard solutions are used for instrument calibration, as noted in Sections 9.1.1 and 9.1.2.

Section 9.1.2: Concentration of the five standard solutions used in calibration are 0.0 (a blank from Section 9.1.1), 0.0002, 0.0005, 0.001, and 0.002 ppm, based on the stock standard preparation method in LS-RMA-4501, and the calibration preparation methods in Section 9.1.2.

Section 9.5: Duplicate analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.

Matrix spike analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.

SW-846 methods, on which many Enseco methods are based, do not contain control limits for matrix spike and replicate analysis. The SW-846 methods do however contain limits on calibration checks. These checks are ±20 percent or greater, thus indicating that the accuracy limit on a matrix spike can never be greater than 80 to 120 percent.

Enseco believes that the control limits indicated in QAPjP Appendix A-11 are realistic, based on the information in the methods and internally developed quality assurance data.

Section 11.1: An example of the data reporting package is provided in QAPjP Appendix C-3.

STANDARD
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PROCEDURE

Subject or Title: Page 1 of 12

Mercury Preparation and Analysis
(Aqueous, Soil, Waste and Leachate Matrices)

SOP No.:	Revision No.:	Effective Date:
LM-RMA-2005	2.0	March 2, 1992

Supersedes: 1.0

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1. Scope and Application

1.1 Analytes

This method is the preparation and cold vapor analysis of aqueous samples (dissolved & total), Cam Wet leachates, EP Toxicity leachates (EPI & EPII), Toxicity Characteristic Leaching Procedure leachates (TCLP), soil samples, industrial waste samples, and oily TCLP extracts for total mercury (organic and inorganic) by cold vapor atomic absorption spectrophotometry (CVAAS).

Prepared By: Chuck Dwyer	Date: 3/2/92
Management Approval: Roger Fuji	Date: 3/5/92
QA Officer Approval: Willie J. Hasse	03/05/92

1.2 Reporting Limits

The reporting limit for mercury (Hg) in both total and dissolved aqueous samples is 0.00020 mg/L. EP Tox, TCLP and Cam Wet leachates are reported at 0.0020 mg/L. Soil or waste samples and oily TCLP extracts are reported at 0.10 mg/kg.

1.3 Applicable Matrices

This method is applicable to total and dissolved aqueous samples, leachates, soil samples and industrial waste matrices.

1.4 Dynamic Range

The dynamic range of samples reported for Hg by CVAA extends upward from a 0.0002 ppm Hg initial concentration.

1.5 Analysis Time

The preparation and analysis time for one batch of 20 samples and required QC is approximately 8 hours (not including instrument warm up time).

2. Method Summary

2.1 A representative aliquot of sample is weighed into a 300 mL biochemical oxygen demand (BOD) bottle. Concentrated sulfuric acid (H_2SO_4), concentrated nitric acid (HNO_3), potassium permanganate ($KMnO_4$), and potassium persulfate ($K_2S_2O_8$) is added. The sample is placed into a water bath and heated to 95°C.

2.1.1 Dissolved and total aqueous samples require a 100 g aliquot (1x).

2.1.2 Leachates require a 10 g aliquot that is diluted to a 100 mL volume with Milli Q water (10x).

2.1.3 Soil or industrial waste samples and oily extracts require a 0.2 g aliquot that is diluted to a 100 mL volume with Milli Q water (500x).

NOTE: If a sample reading for Hg extends beyond the linear range of the highest instrument calibration standard during the first analysis, it must be re-prepped, diluted accordingly and re-analyzed.

2.2 The sample is cooled and sodium chloride-hydroxylamine hydrochloride is added.

2.3 The Hg is then reduced to the elemental state with stannous chloride and aerated from the solution into a closed system. The Hg vapor is passed through a cell positioned in the light path of an atomic absorption spectrometer. The Hg concentration is determined as a function of absorbance at 253.7 nm by CVAAS.

3. Comments

3.1 Interferences

3.1.1 The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transferring, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

3.1.2 Failure to homogenize the sample well prior to removal of the desired aliquot may yield erroneous results.

3.1.3 Sea waters, brines, and industrial effluents high in chlorides require additional permanganate. The chlorides are converted to free chlorine, which also absorbs at 253.7 nm and may cause a positive interference. When this interference is suspected use up to 25 mL of additional potassium permanganate during preparation of the sample. During analysis the head space of the BOD must be purged before the addition of the stannous sulfate.

3.1.4 The recovery of Hg from spiked a sample may be affected if the sample contains concentrations of copper > 10 mg/L.

3.1.5 Concentrations of sodium sulfide > 20 mg/L may may interfere with the recovery of added inorganic Hg from ASTM Type II water.

3.2 Helpful Hints

3.2.1 Bring soil samples within 0.01 g of the desired mass and aqueous samples within 0.2 g of the desired mass.

3.2.2 Visual inspection of the Hg vapor cell and pump tubing before starting the instrument warm up procedure is advised.

3.2.3 DO NOT leave the aerator soaking in 1% HNO_3 after instrument shutdown.

3.2.4 Leave the peristaltic pump on for 15 minutes after instrument shutdown.

4. Safety Issues

4.1 Because mercury vapor is toxic, precaution MUST be taken to avoid its inhalation.

4.2 Proper protective gear (i.e., safety glasses, face shield, gloves, and rubber apron) should be worn when using acids.

4.3 Sample transfers, digestions, and analysis should take place in a properly vented hood, and spills should be cleaned up immediately.

4.4 Sulfuric acid, nitric acid, and stannous chloride are corrosive and will cause skin irritation. Avoid contact with eyes, skin and clothing. Wash thoroughly with copious amounts of water.

4.5 All waste generated from the test will be placed into an approved, correctly labelled waste container.

4.6 After analysis, allow high level samples to aerate in the hood until the analyzer indicates the Hg level has returned to the base line.

4.7 The RMAL safety manual should be referred to for more detailed cautions and instructions.

5. Sample Collection, Preservation and Holding Times

5.1 Samples should be collected in glass or plastic containers.

5.2 Dissolved and total aqueous samples should be acidified with nitric acid to pH <2 at the time of collection.

5.3 For dissolved metals analyses, the samples should be filtered through 0.45 micron filter paper before acid preservation. Filtration must be done in the field or within 24 hr of collection.

5.4 Mercury has a 28 day holding time. All other metals analyses have 180-day holding times.

6. Apparatus

6.1 Hot plate, capable of 95°C.

6.2 Miscellaneous pipettes.

6.3 Miscellaneous benchsheets.

6.4 Calculator.

6.5 300 mL Wheaton biochemical oxygen demand (BOD) bottles.

6.6 Top-loading balance, 0.01 g.

6.7 Water bath with temperature controlled at 95°C.

6.8 Spectro-Products HG-3 cold vapor mercury analyzer.

6.9 Milli volt (mV) meter capable of digital voltage display.

6.10 Peristaltic air pump with variable speed control set at a flow rate of 1 liter/min.

6.11 Aeration tube (a glass frit with coarse porosity attached to air pump).

6.12 250 mL beaker.

7. Reagents and Standards

7.1 ASTM Type II water, (Milli Q or equivalent).

7.2 Hg standard stock solution "A": See RMAL Hg LCS + Calibration Stock SOP LS-RMA-4501.

7.3 Hg DCS stock solution "B": See RMAL Hg LCS + Calibration Stock SOP LS-RMA-4501.

7.4 Sulfuric acid, (H_2SO_4), concentrated, trace metal grade.

7.5 Sulfuric acid, 0.5 N: Carefully add 14 mL of concentrated sulfuric acid to 1 liter of Milli Q water.

7.6 Nitric acid, (HNO_3), concentrated, trace metal grade.

7.7 Nitric acid, (HNO_3), 1% solution (v/v): Carefully add 20 mL concentrated nitric acid to 1980 mL water.

7.8 Potassium permanganate, mercury-free, 5% solution (w/v): dissolve 100 g of potassium permanganate in 2 liters of Milli Q water.

7.9 Potassium persulfate, 5% solution (w/v): dissolve 100 g of potassium persulfate in 2 liters of Milli Q water.

7.10 Sodium chloride-hydroxylamine hydrochloride solution: dissolve a 240 g portion of sodium chloride and 240 g of hydroxylamine hydrochloride in 2 liters of Milli Q water.

7.11 Stannous chloride: add 100 g stannous chloride to 1 liter of 0.5 N H_2SO_4. This mixture is a suspension (should appear cloudy) and should be stirred continuously during use.

8. Procedure

8.1 Instrument Set-up and Calibration

8.1.1 Turn on hot plate, set for 95°C.

8.1.2 Verify the calibration of the balance and pipettes.

8.1.3 Turn on the Hg analyzer and warm up instrument as follows:

8.1.3.1 Turn on the mV meter (0.000 mV will display) and depress the power switch (the button will glow orange) of the Hg analyzer.

8.1.3.2 Rotate the "B" lamp control clockwise until the mV reading is 0.950 mV and stop to allow the Fe reference lamp a 15 minute warm up period.

8.1.3.3 Continue to stage the the mV reading of the reference lamp to 1.000 mV by increasing the previous mV reading by 50% and a 5 minute warm up period.

8.1.3.4 With the reference lamp at 1.000 mV, depress the AGC switch (the button will glow green and a temporary fluctuation in the mV reading will occur, if the mV reading does NOT return to 1.000 mV within 1 minute consult your supervisor for corrective action). After the mV reading stabilizes at 1.000 mV, rotate the "A" control clockwise until the mV reading of the Hg base lamp is 0.500 mV and allow a 15 minute warm up period.

8.1.3.5 Continue to stage the mV reading of the base lamp to 0.000 mV by decreasing the previous mV reading by 50% and a 5 minute warm up period.

NOTE: Reverse the above steps (with exception to the warm up periods) to shut the instrument down.

8.2 General Preparation

8.2.1 Identify the test to be preformed by using the backlogs.

8.2.2 Check for special instructions concerning each sample.

8.2.3 Sign out samples from sample receiving.

8.2.4 Complete bench sheet.

NOTE: Aqueous samples reported in mg/L may NOT be placed on the same bench sheet with soil, waste or oily TCLP extract samples that are reported in mg/kg.

8.2.5 Update the backlog to reflect which samples are in progress.

NOTE: Consult your supervisor if a sample can NOT be located.

8.2.6 Transfer a homogenized (well-mixed) sample aliquot into a clean numbered 300 mL BOD bottle. Cap the BOD.

8.2.6.1 Total and dissolved water samples are analyzed at a 1x dilution and require a 100 g aliquot.

8.2.6.2 EP Tox, TCLP, and Cam Wet leachates are analyzed at a 10x dilution and require a 10 g sample aliquot brought to a 100 g mass with Milli Q water.

NOTE: A sample spike must be analyzed on 1 sample for each batch of EP Tox leachates prepared for Hg.

8.2.6.3 Soil or waste samples and oily TCLP extracts are analyzed at a 500x dilution and require a 0.2 g sample aliquot brought to a 100 g mass with Milli Q water.

8.2.7 Add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 and mix after each addition. Recap the BOD and allow to stand for 5 minutes or until the acid fumes subside.

8.2.8 Add 15 mL potassium permanganate solution. Additional permanganate may be required. Recap BOD. Shake and add additional portions of permanganate as required until the purple color persists for at least 15 min. Recap BOD.

8.2.9 Add 8 mL of potassium persulfate to each bottle and heat for 2 hours in a water bath at 95°C. Remove samples from water bath and allow to cool.

8.3 Analysis

8.3.1 Add 6 mL of sodium chloride-hydroxylamine hydrochloride to the cooled samples in the BOD bottles. This reduces the excess

permanganate and causes the solution to decolorize. Allow the samples to stand with the caps ajar for 5-10 minutes to allow any chlorine gas to dissipate. Treat the samples separately at this point.

8.3.2 Samples are to be analyzed according to the bench sheet in the following order: instrument calibration standards, initial calibration verification (ICV), initial calibration blank (ICB), PB, 2 duplicate control samples (DCS), 10 or less samples, followed by a continuing calibration verification (CCV) and a continuing calibration blank (CCB). For every twenty (or less) samples analyzed, 2 DCS samples will be analyzed along with a CCV and CCB for every 10 samples (or less) to be analyzed.

8.3.3 Insert the aerator into the sample BOD (above the fluid level) and purge the head space for 30 seconds.

8.3.4 Add 5 mL of stannous chloride and immediately attach the aeration apparatus to the BOD bottle. As the Hg is bubbled out of the sample, the absorbance (peak height measured in mV) will rise to a maximum value and level off within approximately 30 seconds. Record the mV reading of each sample on the bench sheet. For all samples reading above 0.05 mV, allow the apparatus to aerate with the frit tube ajar until the reading has returned to the base line (this dissipates the Hg vapors into the hood).

> NOTE: Samples reading above the highest instrument calibration standard must be flagged during analysis. Upon conclusion of the analysis, these BODs will be flushed with hot tap water, rinsed and filled with 275 mL of DI water, and topped off with H_2SO_4 then allowed to stand for 1 hour. The BOD will then be ready for routine washing procedures.

8.3.5 Place the aerator into 100 mLs of 1% HNO_3 and allow to bubble rinse until the next sample analysis.

8.3.6 Analyze the initial calibration standards. Construct a calibration curve by plotting the peak height in mV versus Hg concentration. The linear regression of these data should produce a correlation coefficient for the curve of >0.995.

The initial calibration standards must then be re-analyzed if the calibration curve does not meet the above criterion.

8.3.7 Analyze each sample (including all QC samples) and determine the Hg concentration in ppm from the linear regression fit of the calibration curve.

9. QA/QC Requirements

9.1 Instrument Calibration Standards Preparation

9.1.1 Prepare and analyze a 100 g Milli Q water sample per the procedure as described in section 8.2-8.3, for the instrument calibration blank.

9.1.2 Add, 20 ug, 50 ug, 100 ug, and 200 ug of the Hg standard stock solution "A" to 4 separate BOD bottles, each containing 100 g of Milli Q water, and prepare and analyze the instrument calibration standards as described in section 8.2-8.3.

NOTE: These 5 instrument calibration standards will be used to determine the linear regression of the standard curve. One set of instrument calibration standards will be prepared for each batch of samples prepped.

9.2 ICBs and CCBs must be prepared and analyzed.

9.2.1 ICB: Prepare and analyze a 100 g Milli Q water sample per the procedure described in section 8.2-8.3. One ICB is prepared for each sample batch. It must be analyzed after the ICV sample.

9.2.2 CCB: Prepare and analyze a 100 g Milli Q water sample as per the procedure described in section 8.2-8.3. One CCB shall be prepared for each 10 samples digested or for each batch, whichever is more frequent. It must be analyzed after each CCV sample.

9.3 ICVs and CCVs must be prepared and analyzed.

9.3.1 ICV: Add 200 ug Hg EPA ICV 5 concentrate standard to 100 g of Milli Q water. Prepare and analyze the ICV per the procedure described in section 8.2-8.3. One shall be prepared for each each batch of samples prepped. Recovery must be within 90% to 110% of the true value following QA/QC requirements.

9.3.2 CCV: Add 100 ug Hg standard stock solution "A" to 100 g of Milli Q water. Prepare and analyze the CCV per the procedure described in section 8.2-8.3. One CCV shall be prepared for each 10 (or less) samples digested. Recovery must be within 90% to 110% of the true value following QA/QC requirements.

9.4 DCS samples shall be prepared and analyzed in duplicate for each 20 (or less) samples digested. These 2 samples are prepared by adding 100 ug of the Hg DCS stock solution "B" to 100 g of Milli Q water and digesting them as described in section 8.2-8.3. The recovery for each DCS must be 75% to 125% and their precision must be a relative percent difference of <20% per QA/QC requirements. If this is not the case, terminate the analysis and prep all the associated samples again.

9.5 Client-specific sample duplicates and sample spikes (i.e., matrix spikes and spike duplicates) will be prepared only upon client request and will then appear on the prep scheduling sheet. The MS and SD samples will be spiked with 100 ug of Hg standard stock solution "A".

10. Calculations

Use a scientific calculator with linear regression capabilities to calculate the standard calibration curve. Calculate from mV to concentration of Hg in the sample as ug/L or mg/L.

11. Reporting Requirements

11.1 Total and recoverable aqueous sample results are reported as mg/L units at 0.00020 ppm Hg.

11.2 Leachate sample results are reported in mg/L units at 0.0020 ppm Hg.

11.3 Soil, waste or oily TCLP extract sample results are reported in mg/kg units at 0.10 ppm Hg.

NOTE: Report all results with 2 significant figures if the Hg value is less than 10 units. When the Hg value exceeds 10 units, report all results with 3 significant figures.

12. Review Requirements

12.1 All data packages must be complete, signed, and dated by both the analyst and the peer reviewer. Both the coversheet and the benchsheet should state the analyst's name, the date and type of analysis performed, and the instrument used.

12.2 Benchsheets must contain all calibration information, true values for all QC solutions and recoveries for all QC and analytical spikes.

12.3 Verify that calculations were done correctly by randomly checking a few of them.

13. References

13.1 Methods 7470 and 7471. SW-846 - Test methods for evaluating solid waste. 3rd edition.

13.2 U.S. Environmental Protection Agency Contract Laboratory Program Statement of Work 787 (Rev 12/87) Exhibit D - Analytical Methods.

14. Deviations From Source Methods

14.1 Soils and wastes are digested according to section 7.2 of Method 7471 and are heated in a water bath rather than an autoclave.

STANDARD
OPERATING
PROCEDURE

Subject or Title:
MERCURY LCS & CALIBRATION STOCK

SOP No.:	Revision No.:	Effective Date:
LS-RMA-4501	1.0	04/27/89

Supersedes:
Any SOP's dated prior to 04/27/89

1. TYPE OF STANDARD: Lab. control spike.

1.2. SOLVENT/MATRIX: 5% HNO_3

2. PREPARATION NOTES: To a 500 mL vol. flask add 250 mL DI H_2O plus 25 mL HNO_3. Add 0.5 mL Hg stock. Dilute to volume with DI H_2O. Use serological pipets. Use two sources for each stock.

3. SHELF LIFE: 2 MONTHS.

3.1. Prepare every 8 weeks.

4. SAFETY ISSUES: Refer to the MSDS's

5. COMMENTS: See Lab Procedures SOPS (SOP NO. LP-RMA-0018 thru LP-RMA-0023) for specific lab practices i.e. dilutions, care of glassware, SMI pipets, etc..

Prepared by:
QA Dept.

Date:
04/27/89

Management Approval:

Date:

QA Officer Approval:
Laura Biagnoris

Date:
4/28/89

SOP No.:	Revision No.:	Effective Date:
LS-RMA-4501	1.0	04/27/89

5. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
Mercury (1000 ug/mL)	1.00 mg/L

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-6
SOP Number: LM-RMA-1025
Title: Anion Analysis by Ion Chromatography

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

Section 9.1.4: Duplicate analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.

Section 9.1.5: Matrix spike analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.

SW-846 methods, on which many Enseco methods are based, do not contain control limits for matrix spike and replicate analysis. The SW-846 methods do however contain limits on calibration checks. These checks are ±20 percent or greater, thus indicating that the accuracy limit on a matrix spike can never be greater than 80 to 120 percent. The 80 to 120 percent limits will be utilized.

Section 11.: An example of the data reporting package is provided in QAPjP Appendix C-3.

Subject or Title:

Anion Analysis by Ion Chromatography

SOP No.:	Revision No.:	Effective Date:
LM-RMA-1025	2.0	12/15/90

Supersedes:
1.0 (3/16/88)

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1. Scope and Application

1.1 Analytes

This method is applicable to the determination of the following inorganic anions:

Fluoride	Nitrate (as N)
Chloride	Ortho-Phosphate (as P)
Bromide	Sulfate

Prepared by: Roxanne Sullivan	Date: November 19, 1990
Management Approval:	Date: 12/12/90
QA Officer Approval: Dan Rekenbarger	Date: 12/12/90

1.2 Applicable Matrices

1.2.1 This method is applicable to drinking and surface waters as well as mixed domestic and industrial wastewaters.

1.2.2 The method may be applied to nonaqueous matrices by analyzing a deionized water leachate of the sample (SOP LM-RMA-1074 D.I. Leach).

1.3 Reporting Limits

Enseco's standard reporting limits are:

Analyte	Water (mg/L)
Fluoride	0.5
Chloride	3
Bromide	0.1
Nitrate (as N)	0.1
Ortho-Phosphate (as P)	0.5
Sulfate	5

Lower reporting limits may be available under special conditions. Consult the laboratory for details.

Nonaqueous reporting limits, when determined using DI leach SOP LM-RMA-1074, will be a factor of 5 higher than the above aqueous limits.

1.4 Dynamic Range

The dynamic range of the method is anion specific as indicated in the following table and can be extended by diluting the sample:

Analyte	Dynamic Range (mg/L)	Approximate Retention Times
Fluoride	0.5 - 50	1.0
Chloride	3 - 500	1.6
Nitrite	-------	<1.8
Bromide	0.1 - 50	2.8
Nitrate	0.1 - 50	3.2
Ortho-phosphate	0.5 - 50	5.9
Sulfate	5 - 500	6.5

1.5 Analysis Time

The approximate analysis time for water samples is 10 minutes, not including data reduction. Nonaqueous samples require a DI leach, whose approximate preparation time is 2 hours.

2. Method Summary

A small volume of sample is introduced into the Dionex ion chromatograph. The anions of interest are separated and measured using a system comprised of a guard column, separator column, suppressor column, and conductivity detector. Results are processed using Dionex software.

3. Comments

3.1 Interferences

3.1.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems.

3.1.2 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

3.1.3 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

3.2 Helpful Hints

3.2.1 The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can be eliminated by the addition of the equivalent of 1 mL of concentrated eluent (7.3 100X) to 100 mL of each standard and sample.

3.2.2 Large concentrations of any one ion (such as chloride in brines) may affect the retention time of the other ions and mask peaks.

3.2.3 Molybdenum has the same retention time as sulfate; nitrite elutes very close to chloride.

3.2.4 This method is not recommended for leachate samples when acetic acid is used for pH adjustment. The retention times of the anions differ when large amounts of acetate are present.

3.2.5 This method is not recommended for fluoride. Any anion that is not retained by the column will elute in the area of fluoride and interfere.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco-RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any question or safety concerns, see your supervisor or safety officer.

4.2 Normal accepted laboratory safety practices should be followed during reagent preparation and instrument operation.

5. Sample Collection, Preservation, and Holding Times

5.1 Samples should be collected in scrupulously clean glass or polyethylene bottles.

5.2 Samples are to be preserved and analyzed in accordance with the following:

Analyte	Preservation	Holding Time
Fluoride	None	28 Days
Chloride	None	28 Days
Bromide	4°C	ASAP
Nitrate (as N)	4°C	48 Hrs
O-Phos (as P)	Filter, 4°C	48 Hrs
Sulfate	4°C	28 Days

6. Apparatus

6.1 Balance - Analytical, capable of accurately weighing to 0.0001 g.

6.2 Ion chromatograph - Analytical system complete with ion chromatograph and all required accessories.

6.2.1 Anion guard column: AG4A, 4X50mm, Dionex P/N 37042.

6.2.2 Anion separator column: AS4A, 4X250MM, Dionex P/N 37041.

6.2.3 Anion micromembrane suppressor (AMMS): Dionex P/N 38019.

6.2.4 Detector - Conductivity cell: approximately 6 mL volume.

6.3 Sample bottles: Glass or polyethylene of sufficient volume to allow replicate analyses of anions of interest.

6.4 Miscellaneous laboratory glassware and pipettes.

7. Reagents and Standards

7.1 Reagent water: Distilled or deionized water, free of the anions of interest and containing particles no larger than 0.20 microns.

7.2 Eluent solution: Sodium bicarbonate (CAS RN 144-55-8) 0.0025M, sodium carbonate (CAS RN 497-19-8) 0.00075M. Dissolve 1.0081 g sodium bicarbonate ($NaHCO_3$) and 1.0176 g of sodium carbonate (Na_2CO_3) in reagent water and dilute to 4 liters.

7.3 Regeneration solution (AMMS): Sulfuric acid (CAS RN 7664-93-9) 0.025N. Dilute 2.8 mL conc. sulfuric acid (H_2SO_4) to 4 liters with reagent water.

7.4 Stock standards: May be purchased as certified solutions or prepared from ACS reagent grade materials (dried at 105°C for 30 min) as listed below; may be prepared individually or as one stock standard.

7.4.1 Fluoride (F) 1000 mg/L: Dissolve 2.2100 g sodium fluoride (NaF CAS RN 7681-49-4) in reagent water and dilute to 1 L.

7.4.2 Chloride (Cl) 10,000 mg/L: Dissolve 16.485 g sodium chloride (NaCl CAS RN 7647-14-5) in reagent water and dilute to 1 L.

7.4.3 Bromide (Br) 1000 mg/L: Dissolve 1.288 g sodium bromide (NaBr CAS RN 7647-15-6) in reagent water and dilute to 1 L.

7.4.4 Nitrate (NO_3-N) 1000 mg/L: Dissolve 6.0679 g sodium nitrate ($NaNO_3$ CAS RN 7631-99-4) in reagent water and dilute to 1 L.

7.4.5 Phosphate (PO_4-P) 1000 mg/L: Dissolve 4.3937 g potassium phosphate (KH_2PO_4 CAS RN 7778-77-0) in reagent water and dilute to 1 L.

7.4.6 Sulfate (SO_4) 10,000 mg/L: Dissolve 18.141 g potassium sulfate (K_2SO_4 CAS RN 7778-80-5) in reagent water and dilute to 1 L.

These stock standards are stable for at least one month when stored at 4°C. Dilute working standards should be prepared weekly, except phosphate, which should be prepared fresh daily.

7.5 Working standards: Make up individual working standards for each anion as follows:

Analyte	Concentration (mg/L)	Stock Std. (mLs)	Final Volume (mLs)
Fluoride	100	10	100
Chloride	1000	10	100
Bromide	100	10	100
Nitrate	100	10	100
O-Phos	100	10	100
Sulfate	1000	10	100

7.6 Stock DCS solutions, 1000 ppm.

Stock DCS solutions may be purchased as certified solutions or prepared from ACS reagent grade materials (dried at 105° for 30 minutes) obtained from standards prep as listed below. The stocks may be prepared individually or as one stock DCS. 1000 mg/L DCS stock solutions should be made from a different source than the calibration stocks.

7.7 Working DCS solution

Dilute down the stock DCS solutions into one 100 mL volumetric as follows:

Analyte	Final Concentration (mg/L)	Stock Stds. (mLs)
Fluoride	10	1
Chloride	100	10
Bromide	10	1
Nitrate	10	1
O-Phos	10	1
Sulfate	100	10

7.8 CCV Solution

Prepare a CCV solution at the following concentrations from the working std. solutions in a 100 mL volumetric.

Analyte	Concentration	Working Std. (mLs)
Fluoride	25	2.5
Chloride	250	2.5
Bromide	25	2.5
Nitrate	25	2.5
O-Phos	25	2.5
Sulfate	250	2.5

8. Procedure

8.1 Preparation

8.1.1 Obtain samples from sample receiving. Check each sample for special instructions making copies to put along with the data package. Fill out your benchsheet.

8.1.2 Unless particulate matter is present, water samples are analyzed directly without advanced preparation. Samples containing particulate matter must be filtered through a 0.45 um filter prior to analysis.

8.1.3 Soil samples are prepared by leaching one part sample with five parts deionized water.
See SOP: LM-RMA- 1074 D.I. Leach.

8.2 Instrument Set-up and Calibration

8.2.1 Establish the following ion chromatographic operating parameters:

Columns - AS4A Separator
AGYA Guard
Anion Micromembrane Suppressor

8.2.2 Prepare a series of standards at the following concentrations. Each standard should be made up in 100 mL volumetric with eluent. This is the normal operating curve for Rocky. Other curves can be made to meet client request such as USGS.

Std. 1 is a eluent blank

Std. 2	Concentration (mg/L)	Working/Stock Std. to be diluted (ppm)	mLs
Fluoride	0.1	100	0.1
Chloride	1	1000	0.1
Bromide	0.1	100	0.1
Nitrate	0.1	100	0.1
O-Phos	0.1	100	0.1
Sulfate	1	1000	0.1

Anions by IC

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Std. 3	Concentration (mg/L)	Working/Stock Std. to be diluted (ppm)	mLs
Fluoride	1	100	1
Chloride	10	1000	1
Bromide	1	100	1
Nitrate	1	100	1
O-Phos	1	100	1
Sulfate	10	1000	1

Std. 4	Concentration (mg/L)	Working/Stock Std. to be diluted (ppm)	mLs
Fluoride	10	100	10
Chloride	100	1000	10
Bromide	10	100	10
Nitrate	10	100	10
O-Phos	10	100	10
Sulfate	100	1000	10

Std. 5	Concentration (mg/L)	Working/Stock Std. to be diluted (ppm)	mLs
Fluoride	50	1000	5
Chloride	500	10,000	5
Bromide	50	1000	5
Nitrate	50	1000	5
O-Phos	50	1000	5
Sulfate	500	10,000	5

Eluent - 0.75 m M $NaHCO_3$ (0.0630 g/L)
2.00 m M Na_2CO_3 (0.2120 g/L)

Regenerant -1.025 N H_2SO_4
2.0 mL of 50% H_2SO_4
per 2000 mL

Sample Loop -100 mL

Flow Rate - 2.0 mL/min.

8.2.3 Using injections of 100 uL of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte.

8.2.4 The working calibration curve must be verified on each working day, or whenever the anion eluent is changed, and after every 10 samples. If the response or retention time for any analyte varies from the expected values by more than ± 10%, the test must be repeated, using fresh calibration standards. If the results are still more than ± 10%, an entire new calibration curve must be prepared for that analyte.

8.2.5 Non-linear response can result when the separator column capacity is exceeded (overloading). Maximum column loading (all anions) should not exceed about 5000 ppm.

8.2.6 Linear calibration curve will be used. A study was done using both linear and quadratic calibration and the curves were found to be equivalent.

8.3 Analysis

8.3.1 Check system calibration daily and, if required, recalibrate.

8.3.2 Load and inject a fixed amount of well mixed sample. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.

8.3.3 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for a compound. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

8.3.4 Different dilutions may be required to obtain the concentration of individual anions at the lowest possible reporting limit.

8.3.5 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, spike the sample with an appropriate amount of standard and reanalyze.

8.4 Conclusion

8.4.1 Shut off the I.C. Clean all glassware, apparatus, and the work area.

8.4.2 Return all samples to sample receiving.

8.4.3 Calculate the results according to the calculation section below if not done with the IC software.

8.4.4 Complete the data package making sure it is filled out properly and all results are highlighted. See the attached example. Be sure to include copies of the special instructions. Submit the package to your supervisor for approval.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank is required with every batch of 20 or less samples.

9.1.2 Duplicate laboratory control samples must be analyzed with every batch of 20 or less samples.

9.1.3 Continuing calibration blank (CCB) and continuing calibration verification (CCV) samples must be analyzed after every tenth sample. A CCV and CCB must also be analyzed at the completion of the day's analyses.

9.1.4 Duplicates may be required for project specific QC.

9.1.5 Spikes may be required for project specific QC.

9.2 Acceptance Criteria

9.2.1 Blanks must be less than two times the reporting limit.

9.2.2 Average DCS requirements are summarized in the following table:

Analyte	Accuracy Control Limits*
Fluoride	88 - 112
Chloride	92 - 108
Bromide	90 - 110
Nitrate	91 - 109
Ortho-Phos	75 - 125
Sulfate	93 - 107

9.2.3 The requirements for the RPD for the DCS are summarized in the following table:

Analyte	Precision Control Limits*
Fluoride	15% RPD
Chloride	10% RPD
Bromide	20% RPD
Nitrate	20% RPD
Ortho-Phos	20% RPD
Sulfate	15% RPD

* Subject to change in accordance with Enseco QAPP.

9.2.4 For the DCS data to be considered acceptable, 80% of the acceptance criteria in Sections 9.2.2 and 9.2.3 must be achieved if an IC scan is required.

9.2.5 Standard checks must be within 10% of the expected value.

9.2.6 There are no acceptance criteria for project specific QC.

9.2.7 The calibration curve for each analyte must have a correlation coefficient of 0.995 or greater.

9.3 Corrective Actions

9.3.1 If acceptance criteria described in Section 9.2 are not met, the analysis sequence must be terminated until the source of the problem can be identified and corrected.

9.3.2 If the blank is more than two times the reporting limit, the entire batch of samples must usually be reprepped/reanalyzed or anomalized. Consult your supervisor.

9.3.3 Follow the corrective actions outlined in the current Enseco QAPP Manual.

10. Calculations

10.1 Prepare separate calibration curves for each anion of interest by plotting peak size in area or peak height units of standards against concentration values.

10.2 Compute sample concentration by comparing sample peak response with the standard curve.

10.3 Calculations can be done automatically using the Nelson software.

11. Reporting Requirements

11.1 Units

Report results in mg/L for aqueous samples and mg/kg for soil samples.

11.2 Limits

11.2.1 The reporting limits for the target anions are listed in the following table:

Analyte	Reporting Limit Water (mg/L)
Fluoride	0.5
Chloride	3
Bromide	0.1
Nitrate	0.1
Ortho-Phos	0.5
Sulfate	5

11.2.2 If dilutions were required for any reason the reporting limit is multiplied by the dilution factor.

11.2.3 If the blank shows contamination (but less than two times the reporting limit) the reporting limit is raised to the blank value.

11.3 Significant Figures

11.3.1

Range	Sig. Figures
< 10	2
10	3
> 10	3

11.3.2 Observed values below the detection limit must be reported as not detected (ND).

11.4 LIMS Data Entry

The usual standards for data entry apply.

11.5 Document any non-standard procedure or any anomalies.

12. Review Requirements

12.1 All data packages must be complete. They must be properly signed and dated.

12.2 Confirm that the special instructions were appropriately observed.

12.3 Check any calculations.

12.4 Check the reporting limits. The standard reporting limit must be multiplied by any dilution factor or raised to any blank contamination value.

12.5 Verify that the results were correctly anomalized.

13. References

13.1 Method Source

This method is based on EPA Test Method 300.0 (Method A) entitled, "The Determination of Inorganic Anions in Water by Ion Chromatography," December 1989.

13.2 Related Documents

13.2.1 Enseco QAPP

13.2.2 M-EQA-0002 Internal QC checks--Laboratory Performance QC

13.2.3 LM-RMA-1074 D.I. Leach

13.3 Deviations from Source Method and Rationale

13.3.1 The columns specified in the referenced method have been substituted. This change was motivated by advances in the technology which occurred after publication of the method.

13.3.2 Although the source method does not recommend fluoride analysis by IC it will be used occasionally by an experienced chromatographer knowledgable in the particular problems.

13.3.3 Nitrite has been deleted from the list of target anions because it elutes so close to chloride.

13.3.4 Enseco's Quality Assurance Plan will be followed instead of the quality program outlined in Method 300.0.

13.4 SOP Updates (Revision 1.0 to Revision 2.0)

13.4.1 Format updated.

13.4.2 Range was extended to 500 mg/L for chloride.

13.4.3 The SOP has been modified to reflect the revised Method 300.0 (Method A), December 1989.

ION CHROMATOGRAPHY

Process File: 112990A.PRN Analyst: Steve P Date: 11-~~30~~ 29-90 SBP 11-30

UNITS: mg/L Checked & Approved: _____

Sample Name	DF	Chloride (0.2 RPD)	Bromide (1.0 RPD)	Sulfate (1.0 RPD)		
ICB	1	ND(3.0)	ND(0.20)	ND(5.0)		
DCS 1A	1	99.0(99)	19.3(97)	203(102)		
DCS 2A	1	98.8(99)	19.1(96)	201(101)		
12171-07 X50	50	4860	19.1	4810		
12171-07 X10	10	OverRange	224	ND(50)		
BLANK	1	ND(3.0)	ND(0.20)	5.4		
12171-08 X25	25	3600	10.7	3110		
12171-08 X5	5	31.2	9.8	OverRange		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
12171-09 X50	50	3540	11.1	2960		
12171-09 X10	10	OverRange	11.4	3240		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
12171-10 X50	50	4100	11.5	4080		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCB	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCV	1	102(102)	9.5(95)	98.9(99)		
12171-10 X10	10	OverRange	11.5	4430		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
12470-01 X2	2	43.7	0.80	ND(10)		
SRP 12470-01	1	45.6	0.80	ND(5.0)		
12470-03 X5	5	73.1	ND(1.0)	1410		
12470-03	1	79.2	0.47	ND(5.0)		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
12470-05 X10	10	138	ND(2.0)	2740		
12470-05	1	152	1.3	ND(5.0)		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCB	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCV	1	100(100)	9.2(92)	97.1(97)		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
ICB	1	ND(3.0)	ND(0.20)	ND(5.0)		
DCS 1B	1	105(105)	20.0(100)	212(106)		
DCS 2B	1	102(102)	19.6(98)	206(103)		
12470-07 X5	5	65.1	ND(1.0)	ND(25)		
12470-07	1	66.8	0.33	ND(5.0)		
12470-08 X5	5	65.6	ND(1.0)	ND(25)		
12470-08	1	67.9	0.33	ND(5.0)		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
12429-01 X2	2	11.3	ND(0.40)	571		
12429-01	1	10.9	ND(0.20)	OverRange		
12432-01	1	ND(3.0)	ND(0.20)	33.4		
12432-02	1	ND(3.0)	ND(0.20)	33.7		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCB	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCV	1	105(105)	9.6(96)	100(100)		
12432-04 X5	5	ND(15)	ND(1.0)	2270		
12432-04	1	8.1	ND(0.20)	ND(5.0)		
BLANK	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCB	1	ND(3.0)	ND(0.20)	ND(5.0)		
CCV	1	104(104)	9.5(95)	101(101)		

At-A-Glance Summary

EPA

Test Method
The Determination of Inorganic Anions
in Water by Ion Chromatography-Method 300.0

Revised Dec. 1989

Method A

Column Specified:	Dionex AS4A		
Detection Specified:	Dionex, Chemically Suppressed Conductivity Detector		
Ions Specified:	Bromide Chloride	Nitrate Nitrite	Ortho-phosphate Sulfate
Validated For:	Reagent Water Drinking Water	Surface Water Waste Water	Ground Water Solid Extract

Note: Currently approved for compliance monitoring of nitrate in drinking water. Recommended for approval for compliance monitoring of nitrite and sulfate when these contaminants are regulated in drinking water.

Method B

Column Specified:	Dionex AS9		
Detection Specified:	Dionex, Chemically Suppressed Conductivity Detection		
Ions Specified:	Bromate	Chlorate	Chlorite
Validated For:	Raw Water	Drinking Water	

Note: Recommended for approval for compliance monitoring when these contaminants are regulated.

2.2 The main differences between Method A and B are the separator columns, guard columns and eluents. Sections 6 and 7 will elicit the differences.

2.3 In order to use this method for solids an extraction procedure must be performed (See 11.7).

3. Definitions

3.1 Stock standard solution - a concentrated solution containing a single certified standard that is a method analyte. Stock standard solutions are used to prepare calibration standards.

3.2 Calibration standards (CAL) - a solution of analytes prepared in the laboratory from stock standard solutions and diluted as needed and used to calibrate the instrument response with respect to analytic concentration.

3.3 Quality control sample (QCS) - a solution containing known concentrations of analytes, prepared by a laboratory other than the laboratory performing the analysis. The analyzing laboratory uses this solution to demonstrate that it can obtain acceptable identifications and measurements with a method.

3.4 Performance evaluation sample (PE) - a solution of method analytes distributed by the Quality Assurance Research Division (QARD), Environmental Monitoring Systems Laboratory (EMSL-Cincinnati), USEPA, Cincinnati, Ohio, to multiple laboratories for analysis. A volume of the solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses used by the QARD to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst. Analyte true values are unknown to the analyst.

3.5 Laboratory performance check standards (LPC) - a solution of analytes prepared in the laboratory by adding appropriate volumes of the stock standard solutions to reagent water used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

3.6 Laboratory duplicates (LD) - two aliquots of the same sample that are treated exactly the same throughout laboratory analytical procedures. Analyses of laboratory duplicates indicate precision associated with laboratory procedures but not the sample collection, preservation, or storage procedures.

3.7 Field duplicates (FD) - two samples taken at the same time and placed under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

3.8 Laboratory fortified sample matrix (LFM) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

4. Interferences

4.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems.

4.2 The water dip or negative peak that elutes near and can interfere with the fluoride peak can usually be eliminated by the addition of the equivalent of 1 mL of concentrated eluent (7.3 100X) to 100 mL of each standard and sample.

4.3 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

4.4 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

4.5 This method is not recommended for fluoride. Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L this interference may not be significant, however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.6 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

5. Safety

5.1 Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. No known carcinogenic materials are used in this method.

6. Apparatus and Materials

6.1 Balance - Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2 Ion chromatograph - Analytical system complete with ion chromatograph and all required accessories including syringes, analytical columns, compressed gasses and detectors.

6.2.1 Anion guard column: A protector of the separator column. If omitted from the system the retention times will be shorter. Usually packed with a substrate the same as that in the separator column.

6.2.2 Anion separator column: This column produces the separation shown in Figures 1 and 2.

6.2.2.1 Anion separator column (Method A): The separation shown in Figure 1 was generated using a Dionex AS4A column (P/N 37041). An optional column may be used if comparable resolution of peaks is obtained, and the requirements of section 10.2 can be met.

6.2.2.2 Anion separator column (Method B). The separation shown in Figure 2 was generated using a Dionex AS9 column (P/N 42025). An optional column may be used if comparable resolution of peaks is obtained and the requirements of section 10.2 can be met.

6.2.3 Anion suppressor device: The data presented in this method was generated using a Dionex Anion Micro-Membrane Suppressor (P/N 37106).

6.2.4 Detector - Conductivity cell: approximately 1.25 µL internal volume, (Dionex, or equivalent) capable of providing data as required in section 10.2.

6.3 The Dionex AI-450 Data Chromatography Software (Version 2.0) was used to generate all the data in the attached tables. Systems using a stripchart recorder and integrator or other computer based data system may achieve approximately the same MDL's but the user should demonstrate this by the procedure outlined in Section 10.2.

7. Reagents and Consumable Materials

7.1 Sample bottles: Glass or polyethylene of sufficient volume to allow replicate analyses of anions of interest.

7.2 Reagent water: Distilled or deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

7.3 Eluent solutions:

7.3.1 Eluent solution (Method A): Sodium bicarbonate (CAS RN 144-55-8) 1.7 mM, sodium carbonate (CAS RN 497-19-8) 1.8 mM. Dissolve 0.2856 g sodium bicarbonate ($NaHCO_3$) and 0.3816 g of sodium carbonate (Na_2CO_3) in reagent water (7.2) and dilute to 2 liters.

7.3.2 Eluent solution (Method B): Sodium bicarbonate (CAS RN 144-55-8) 0.2 mM, sodium carbonate (CAS RN 497-19-8) 1.4 mM. Dissolve 0.0336 g sodium bicarbonate ($NaHCO_3$) and 0.2968 g of sodium carbonate (Na_2CO_3) in reagent water (7.2) and dilute to 2 liters.

7.4 Regeneration solution (MicroMembrane Suppressor): Sulfuric acid (CAS RN-7664-93-9) 0.025N. Dilute 2.8 mL conc. sulfuric acid (H_2SO_4) to 4 liters with reagent water.

7.5 Stock standard solutions, 1000 mg/L (1 mg/ml): Stock standard solutions may be purchased as certified solutions or prepared from ACS reagent grade materials (dried at 105°C for 30 min.) as listed below.

7.5.1 Bromide (Br^-) 1000 mg/L: Dissolve 1.2876 g sodium bromide (NaBr, CAS RN 7647-15-6) in reagent water and dilute to 1 liter.

7.5.2 Bromate (BrO_3^-) 1000 mg/L: Dissolve 1.3057 g of potassium bromate ($KBrO_3$, CAS RN 7758-01-2) in reagent water and dilute to 1 liter.

7.5.3 Chlorate (ClO_3^-) 1000 mg/L: Dissolve 1.2753 g sodium chlorate ($NaClO_3$, CAS RN 7775-09-9) in reagent water and dilute to 1 liter.

7.5.4 Chloride (Cl^-) 1000 mg/L: Dissolve 1.6485 g sodium chloride (NaCl, CAS RN 7647-14-5) in reagent water and dilute to 1 liter.

7.5.5 Chlorite (ClO_2^-) 1000 mg/L: Dissolve 1.3410 g of sodium chlorite ($NaClO_2$, CAS RN 7758-19-2) in reagent water and dilute to 1 liter.

7.5.6 Nitrate (NO_3^-–N) 1000 mg/L: Dissolve 6.0679 g sodium nitrate ($NaNO_3$, CAS RN 7631-99-4) in reagent water and dilute to 1 liter.

7.5.7 Nitrite (NO_2^-–N) 1000 mg/L: Dissolve 4.9257 g sodium nitrite ($NaNO_2$, CAS RN 7632-00-0) in reagent water and dilute to 1 liter.

7.5.8 Phosphate (HPO_4^{2-}–P) 1000 mg/L: Dissolve 4.3937 g potassium phosphate, monobasic (KH_2PO_4, CAS RN 7778-77-0) in reagent water and dilute to 1 liter.

7.5.9 Sulfate (SO_4^{2-}) 1000 mg/L: Dissolve 1.8141 g potassium sulfate (K_2SO_4, CAS RN 7778-80-5) in reagent water and dilute to 1 liter.

Note: Stability of standards: Stock standards (7.5) are stable for at least one month when stored at 4°C. Dilute working standards should be prepared weekly, except those that contain nitrite and phosphate should be prepared fresh daily.

8. Sample Collection, Preservation and Storage

8.1 Samples should be collected in scrupulously clean glass or polyethylene bottles.

8.2 Sample preservation and holding times for the anions that can be determined by this method are as follow.

Analyte	Preservation	Holding Time
Bromate	None required	28 days
Bromide	None required	28 days
Chlorate	None required	28 days
Chloride	None required	28 days
Chlorite	Cool to 4° C	immed.
Nitrate-N	Cool to 4° C	48 hours
Nitrite-N	Cool to 4° C	48 hours
O-Phosphate-P	Cool to 4° C	48 hours
Sulfate	Cool to 4° C	28 days

8.3 The method of preservation and the holding time for samples analyzed by this method are determined by the anions of interest. In a given sample, the anion that requires the most preservation treatment and the shortest holding time will determine the preservation treatment. It is recommended that all samples be cooled to 4° C and held for no longer than 48 hours for Method A and run as soon as possible for Method B.

9. Calibration and Standardization

9.1 Establish ion chromatographic operating parameters equivalent to those indicated in Table 1A or 1B.

9.2 For each analyte of interest, prepare calibration standards at a minimum of three concentration levels and a blank by adding accurately measured volumes of one or more stock standards (7.5) to a volumetric flask and diluting to volume with reagent water. If the working range exceeds the linear range of the system, a sufficient number of standards must be analyzed to allow an accurate calibration curve to be established. One of the standards should be at a concentration near, but above, the method detection limit if the system is operated on various attenuator ranges. The other standards should correspond to the range of concentrations expected in the sample or should define the working range of the detector. Unless the

attenuator range settings are proven to be linear, each range must be calibrated individually.

9.3 Using injections of 0.1 to 1.0 mL (determined by injection loop volume) of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte. During this procedure, retention times must be recorded.

9.4 The calibration curve must be verified on each working day, or whenever the anion eluent is changed, and after every 20 samples. If the response or retention time for any analyte varies from the expected values by more than ±10%, the test must be repeated, using fresh calibration standards. If the results are still more than ±10%, a new calibration curve must be prepared for that analyte.

9.5 Non-linear response can result when the separator column capacity is exceeded (overloading). The response of the detector to the sample when diluted 1 to 1 and when not diluted should be compared. If the calculated responses are the same, samples of this total anionic concentration need not be diluted.

10. Quality Control

10.1 Each laboratory using this method should have a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability (10.2) and the analysis of fortified samples as a continuing check on performance. The laboratory should maintain performance records to define and document the quality of data that are generated.

10.1.1 In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in Section 10.2.

10.1.2 The laboratory should fortify and analyze a minimum of 10% of all samples to monitor continuing laboratory performance. Field and laboratory duplicates should also be analyzed.

10.2 Before performing any analyses, the analyst should demonstrate the ability to generate acceptable accuracy and precision with this method, using a laboratory performance standard.

10.2.1 Select a representative check concentration for each analyte to be measured. Using stock standards, prepare a laboratory performance check sample concentrate in reagent water 100 times more concentrated than the selected concentrations.

10.2.2 Using a pipet, add 1.00 mL of the check sample concentrate (10.2.1) to each of a minimum of four 100-mL aliquots of reagent water. Analyze the aliquots according to the procedure in Section 11.

10.2.3 Calculate the average percent recovery, (R), and the standard deviation(s) of the percent recovery, for the results.

10.2.4 Using the appropriate data from Table 2, determine the recovery and single operator precision expected for the method, and compare these results to the values calculated in Section 10.2.3. If the data are not comparable within control limits (10.3.1), review potential problem areas and repeat the test.

10.3 The analyst must calculate method performance criteria and define the performance of the laboratory for each spike concentration of analyte being measured.

10.3.1 Calculate upper and lower control limits for method performance as follows:

Upper Control Limit (UCL) = R + 3 s
Lower Control Limit (LCL) = R - 3 s

where R and s are calculated as in Section 10.2.3. The UCL and LCL can be used to construct control charts that are useful in observing trends in performance.

10.4 The laboratory should develop and maintain separate accuracy statements of laboratory performance for each matrix being analyzed by the laboratory. An accuracy statement for the method is defined as R ± s. The accuracy statement should be developed by the analyses of four aliquots of water or wastewater, as described in Section 10.2.2, followed by the calculation of R and s.

10.5 Before processing any samples, the analyst must demonstrate through the analysis of an aliquot of reagent water that all glassware and reagent interferences are under control. Each time there is a change in reagents, a laboratory reagent blank must be processed as a safeguard against laboratory contamination.

10.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists over the identification of a peak in the chromatogram, confirmatory techniques such as sample dilution and fortification, must be used. Whenever possible, the laboratory should perform analysis of quality control check samples and participate in relevant performance evaluation sample studies.

10.7 In order to verify that standards have been prepared correctly a reference standard check should be performed using a standard of known concentration prepared by an independent source.

10.8 With each batch of samples processed analyze a single laboratory fortified blank containing each analyte of concern at a concentration at or near those used in the reagent water data in Tables 2A or 2B. If more than 20 samples are run in a batch analyze one LFB for every 20 samples. Evaluate the accuracy by compairing to Tables 2A of 2B. If acceptable data cannot be obtained, locate the problem and correct it.

10.9 At least quarterly, replicates of LFBs should be analyzed to determine the precision of the laboratory measurements. Add these results to the ongoing control charts to document data quality.

10.10 When using Part B, the analyst should be aware of the purity of the reagents used to prepare standards. Allowances must be made when the solid materials are less than 99% pure.

11. Procedure

11.1 Tables 1A and 1B summarizes the recommended operating conditions for the ion chromatograph. Included in this table are estimated retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the requirements of Section 10.2 are met.

11.2 Check system calibration daily and , if required, recalibrate as described in Section 9.

11.3 Load and inject a fixed amount of well mixed sample. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.

11.4 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

11.5 If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

11.6 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

Note: Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases this peak migration may produce poor resolution or identification.

11.7 The following extraction should be used for solid materials. Add an amount of reagent water equal to ten times the weight of dry solid material taken as a sample. This slurry is mixed together for ten minutes using a magnetic stirring device. Filter the resulting slurry before injecting using a 0.45 μ membrane type filter. This can be the type that attaches directly to the end of the syringe. Care should be taken to show that good recovery and identification of peaks is obtained with the users matrix through the use of spikes.

12. Calculation

12.1 Prepare separate calibration curves for each anion of interest by plotting peak size in area, or peak height units of standards against concentration values. Compute sample concentration by comparing sample peak response with the standard curve.

12.2 Report results in mg/L.

12.3 Report NO_2^- as N
NO_3^- as N
HPO_4^{2-} as P

13. Precision and Accuracy - Method Detection Limit

13.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations listed in Table 1A and 1B were obtained using reagent waters.

13.2 Single operator accuracy and precision for reagent, drinking and surface water, and mixed domestic and industrial wastewater are listed in Table 2A and 2B.

14. References

14.1 "Determination of Inorganic Disinfection By-Products by Ion Chromatography", J. Pfaff, C. Brockhoff, J. Am. Water Works Assoc., Vol 82, No. 4, pg 192.

14.2 Standard Methods for the Examination of Water and Wastewater, Method 4110B, "Anions by Ion Chromatography" proposed for the supplement 17th Edition of Standard Methods.

14.3 Dionex, System 4000 Operation and Maintenance Manual, Dionex Corp., Sunnyvale, California 94086. 1988.

14.4 Method Detection Limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, Environmental Science and Technology, Vol. 15, Number 12, page 1426, December 1981.

Copies of this method provided courtesy Dionex Corporation.

Table 1A. Chromatographic Conditions and Detection Limits in Reagent Water (Method A)

Analyte	Peak # *	Retention Time (min)	MDL (mg/L)
Chloride	2	1.7	0.02
Nitrite-N	3	2.0	0.004
Bromide	4	2.9	0.01
Nitrate-N	5	3.1	0.002
O-Phosphate-P	6	5.3	0.003
Sulfate	7	6.8	0.02

Standard Conditions:

Columns: as specified in 6.2.2.1
Detector: as specified in 6.2.4
Eluent: as specified in 7.3.1

Pump Rate: 2.0 mL/min.
Sample Loop: 50 µL

MDL calculated from data system using a y-axis selection of 1000 ns and with a stripchart recorder with an attenuator setting of 1 uMHO full scale. Other settings would produce an MDL proportional to their value.

* See figure 1

Table 1B. Chromatographic Conditions and Detection Limits in Reagent Water (Method B)

Analyte	Peak # *	Retention Time (min)	MDL (mg/L)
Chlorite	1	2.8	0.01
Bromate	2	3.2	0.02
Chlorate	3	7.1	0.003

Standard Conditions:

Column: as specified in 6.2.2.2
Detector: as specified in 6.2.4
Eluent: as specified in 7.3.2

Pump Rate: 1.0 mL/min.
Sample Loop: 50 µL
Attentuation - 1
y - axis - 500 ns

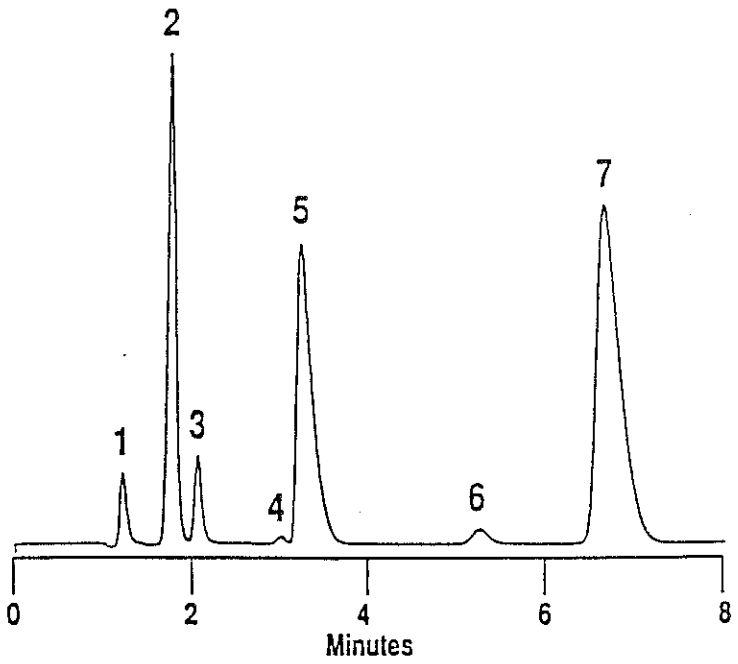
*See figure 2

able 2A. Single-Operator Accuracy and Precision of Standard Anions (Method A)

naly	Sample Type	Spike (mg/L)	Number of Replicates	Mean Recovery %	Standard Deviation (mg/L)
romide	RW	5.0	7	99	0.08
	DW	5.0	7	105	0.10
	SW	5.0	7	95	0.13
	WW	5.0	7	105	0.34
	GW	5.0	7	92	0.34
	SD	2.0	7	82	0.06
hloride	RW	20.0	7	96	0.35
	DW	20.0	7	108	1.19
	SW	10.0	7	86	0.33
	WW	20.0	7	101	5.2
	GW	20.0	7	114	1.3
	SD	20.0	7	90	0.32
trate-N	RW	10.0	7	103	0.21
	DW	10.0	7	104	0.27
	SW	10.0	7	93	0.17
	WW	10.0	7	101	0.82
	GW	10.0	7	97	0.47
	SD	10.0	7	82	0.28
trite-N	RW	10.0	7	97	0.14
	DW	10.0	7	121	0.25
	SW	5.0	7	92	0.14
	WW	5.0	7	91	0.50
	GW	10.0	7	96	0.35
	SD	2.0	7	98	0.08
-Phosphate-P	RW	10.0	7	99	0.17
	DW	10.0	7	99	0.26
	SW	10.0	7	98	0.22
	WW	10.0	7	106	0.85
	GW	10.0	7	95	0.33
	SD	0.0	0	00	0.00
ulfate	RW	20.0	7	99	0.40
	DW	50.0	7	105	3.35
	SW	40.0	7	95	1.7
	WW	40.0	7	102	6.4
	GW	40.0	7	112	3.2
	SD	0.0	0	00	0.0

N = Reagent Water WW = Wastewater
N = Drinking Water GW = Groundwater
N = Surface Water SD = Solid

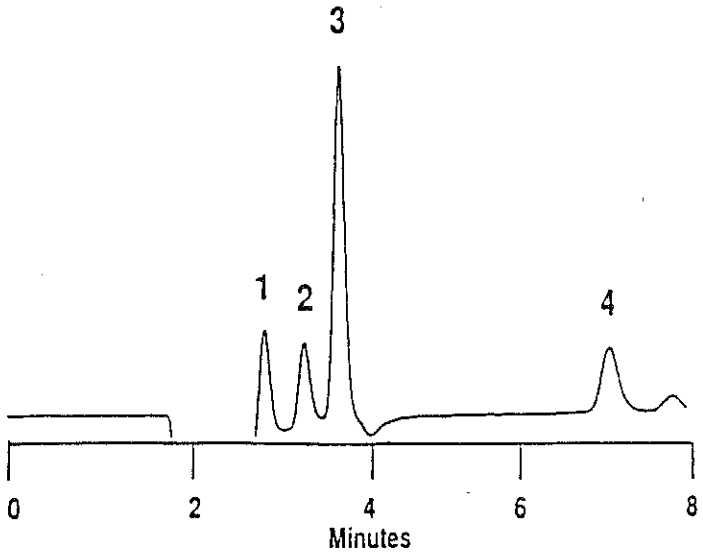
Method A



Peak	Ret. Time	Ion	mg/L
1	1.17	F^-	2
2	1.73	Cl^-	20
3	2.02	NO_2^-	2
4	2.95	Br^-	2
5	3.20	NO_3^-	10
6	5.38	HPO_4^{2-}	2
7	6.92	SO_4^{2-}	60

Figure 1. Chromatogram showing separation using the AS4A column

Method B



Peak	Ret. Time	Ion	mg/L
1	2.75	ClO_3^-	0.1
2	3.23	BrO_3^-	0.1
3	3.63	Cl^-	0.1
4	7.08	ClO_4^-	0.1

Figure 2. Chromatogram showing separation using the AS9 column

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-7
SOP Number: LM-RMA-1079
Title: Ammonia Nitrate + Nitrite TRAACS

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

Section 1.3: Sample distillation will not be used in this ammonia analysis. Distillation is only performed for NPDES projects.

Section 9.1.4: The spiking concentration is 1 mg/L as N after the dilution of 1 ml of Intermediate Standard to 10 ml total volume using the sample. This is due to the Intermediate Sample having a concentration of 10 mg/L of N, as indicated in the preparation described in Sections 7.3.1, 7.3.2, 7.3.3 and 7.3.4 of the SOP.

Section 9.2: Acceptance control limits for blanks are documented in QAPjP Appendix C-1.

A copper/cadmium column will be used in the analysis for the determination of nitrate + nitrite. Calibration standards will be treated in the same way as the environmental samples.

Subject or Title: **Ammonia Nitrate + Nitrite Traacs**

SOP No.: LM-RMA-1079

Revision No.: Original

Effective Date: May 2, 1988

Enseco

Supersedes:

1. Scope and Application

1.1 Analytes

This method is to be used for the analysis of ammonia and nitrate + nitrite.

Nitrite may be analyzed by bypassing the cadmium reduction column.

Nitrate may be determined by subtracting the nitrite result from the nitrate + nitrite result.

1.2 Detection Limit

The detection limit is 0.1 mg/L for both ammonia and nitrate + nitrite, calculated as nitrogen.

Lower concentrations may be analyzed by expanding the scale.

1.3 Applicable Matrices

This method is applicable to the analysis of surface and saline waters, and domestic and industrial wastes. NPDES samples for ammonia must be distilled before analysis.

This method can be used for the analysis of soils and wastes after the appropriate prep is performed.

Prepared by: Date:

Management Approval: Date:

QA Officer Approval: Date:

1.4 Dynamic Range

The appropriate linear range of the method is from 0.1 to 3 mg/L as N for both parameters.

1.5 The approximate analytical time is 10 minutes per sample. Ammonia and nitrate + nitrite are determined simultaneously.

2.0 Summary of Method

Samples are analyzed on the Technical TRAACS system which performs automated colorimetric analyses.

Samples are introduced by means of an automatic sampler, split, and sent to two separate analytical cartridges. One analyzes for ammonia and the other analyzes for nitrate + nitrite.

Ammonia is analyzed using the Berthelot Reaction, forming a blue compound with sodium phenoxide and sodium hypochlorite. The color is intensified by the addition of sodium nitroprusside. EDTA prevents the precipitation of calcium and magnesium hydroxides.

Nitrate is reduced to nitrite at pH 7.5 in a copper/cadmium reductor column. Nitrite then reacts in acidic solution with sulfanilamide to form a diazo compound which then couples with N-1-naphthylethylenediamine dihydrochloride to form a reddish-purple dye. Nitrite originally present in the sample is analyzed together with the reduced nitrate. Nitrite can be determined separately by bypassing the copper/cadmium column.

3. Comments

3.1 Interferences

3.1.1 Calcium and magnesium may precipitate and clog the ammonia channel. EDTA is used to prevent this. This may not be effective on salt water samples.

3.1.2 Color and turbidity in the samples will interfere. Turbidity is removed by filtration. Samples with color interferences must be diluted until no interference is detected.

3.1.3 The samples, standards, and rinse water must all contain the same concentration of acid.

3.1.4 Low nitrate results may be obtained for samples containing iron, copper, and other metals. EDTA is added to eliminate this interference.

3.1.5 Samples containing oil and grease may coat the copper/cadmium column, causing low nitrate results.

3.1.6 Samples for NPDES must be distilled before ammonia analysis. The procedure is given in a separate SOP.

4.0 Safety Issues

Phenol is highly toxic and can be absorbed through the skin or by inhalation of the vapor. Wear gloves and handle concentrated solutions in a hood.

Nitrate + nitrite waste contains cadmium and must not be emptied into the sinks. Ammonia waste contains phenol and should also not be disposed of in the sinks. The two wastes should be segregated, not combined.

5.0 Sample Collection and Preservation

Samples are preserved by adding sulfuric acid (2mL per 1000 ml sample) and refrigerating to 4 C.

6. Apparatus

TRAACS system consisting of sampler, analytical console, and PC/controller.

Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

7.1 Ammonia Analysis

7.1.1 Alkaline Phenol

Add 83 g liquified phenol to about 800 ml deionized water. Cool in an ice bath or under tap water and slowly add 96 g sodium hydroxide solution (50% w/w) with constant mixing. Cool to room temperature, dilute to 1000 ml with deionized water, and mix. Store in an amber glass container and prepare fresh every two weeks.

7.1.2 Sodium Hypochlorite Solution

Dilute 86 ml commercial bleach (5%) to 100 ml with deionized water. Prepare fresh weekly.

7.1.3 Sodium Nitroprusside Solution

Dissolve 1.1 g sodium nitroprusside in about 600 ml deionized water and dilute to 1000 ml with deionized water. Store in an amber container and prepare fresh monthly.

7.1.4 Disodium EDTA

Dissolve 1.0 g 50% w/w sodium hydroxide and 41.0 g disodium EDTA in about 800 ml deionized water. Dilute to 1000 ml, add 3 ml Brij-35, and mix.

7.1.5 0.2% Sulfuric Acid

Carefully add 4.0 ml concentrated sulfuric acid to 2000 ml deionized water and mix.

7.1.6 Rinse Solution

Carefully add 4.0 ml concentrated sulfuric acid to 2000 ml deionized water. Add 3 ml Brij-35 and mix.

7.2 Nitrate + Nitrite Analysis

7.2.1 50% Triton X-100

Place 50 ml isopropanol in a 100 ml glass stoppered graduated cylinder. Add 50 ml Triton X-100, stopper, and mix by inverting several times.

7.2.2 Stock Imidizole Buffer, 0.1 M

Dissolve 6.81 g imidizole in about 900 ml deionized water. Adjust pH to 7.5 +/- 0.05 with hydrochloric acid. Dilute to 1000 ml with deionized water and mix thoroughly.

7.2.3 Copper Sulfate, 0.01 M

Dissolve 2.5 g copper sulfate in deionized water and dilute to 1000 ml.

7.2.4 Copper Sulfate, 0.001 M

Dilute 10 ml 0.01 M copper sulfate to 100 ml with deionized water.

7.2.5 Working Imidizole Buffer, 0.05 M

Add 0.5 ml 0.001 M copper sulfate to 50 ml stock imidizole buffer. Dilute to 100 ml with deionized water, add 0.1 ml 50% Triton X-100 and mix thoroughly. Prepare this solution fresh daily.

7.2.6 Sulfanilamide Solution

Add 50 ml concentrated hydrochloric acid to 600 ml deionized water in a 1000 ml flask. Add 5.0 g sulfanilamide and swirl to dissolve. Dilute to volume with deionized water, add 0.5 ml 50% Triton 100-X, and mix. Store in an amber container and refrigerate when not in use.

7.2.7 NED Solution

Dissolve 0.5 g N-(1-naphthyl)ethylenediamine dihydrochloride in about 600 ml deionized water. Dilute to 1000 ml, add 0.5 ml 50% Triton X-100, and mix thoroughly. Store in an amber container and refrigerate when not in use.

7.2.8 2 N Hydrochloric Acid

Carefully add 165 ml concentrated hydrochloric acid to about 600 ml deionized water with constant mixing. Cool to room temperature, dilute to 1000 ml, add 0.5 ml Triton X-100, and mix.

7.2.9 2 N Nitric Acid

Carefully add 12.5 ml concentrated nitric acid to about 60 ml deionized water with constant mixing. Cool to room temperature and dilute to 100 ml with deionized water.

7.2.10 Copper Activating Solution

Dilute 50 ml 0.01 M copper sulfate to 100 ml with deionized water, add 0.1 ml 50% Triton X-100, and mix.

7.2.11 5 N Sulfuric Acid

Slowly and carefully add 139 ml concentrated sulfuric acid to about 500 ml deionized water with constant mixing. Cool to room temperature and dilute 1000 ml.

7.3 Calibration Standards

7.3.1 Ammonia Stock Standard, 1000 mg/L as N

Dry Ammonium Chloride at 105 C. Dissolve 3.819 g in 600 ml deionized water, add 2 ml concentrated sulfuric acid, and dilute to 1000 ml with deionized water. This solution is to be obtained from standards prep.

7.3.2 Nitrate Stock Standard, 1000 mg/L as N

Dissolve 7.218 g Potassium Nitrate in about 600 ml deionized water. Carefully add 2 ml concentrated sulfuric acid, mix and dilute to 1000 ml with deionized water. This solution is to be obtained from Standards Prep.

7.3.3 Intermediate Mixed Standard, 10 mg/L as N

Pipette 1.0 ml of each Stock Standard into a 100 ml volumetric flask and dilute to volume with 0.2% sulfuric acid.

7.3.4 Working Standards

Prepare dilutions as indicated below. All dilutions should be made with 0.2% sulfuric acid and NOT with deionized water:

Volume Intermediate Standard	Final Volume	Concentration mg/L as N
1.0 ml	100 ml	0.1
2.0 ml	100 ml	0.2
5.0 ml	100 ml	0.5
10.0 ml	100 ml	1.0
20.0 ml	100 ml	2.0
30.0 ml	100 ml	3.0

8. Procedure

8.1 Sample Preparation

8.1.1 NPDES samples for ammonia must be distilled before analysis using the procedure given in the Ammonia Distillation SOP.

8.1.2 Soils must be taken through the D.I. Leach procedure before analysis.

8.1.3 Check the pH of all samples to ensure that they have been properly preserved. If necessary, add sulfuric acid to produce a final concentration of 0.2%.

8.2 Calibration

Standards are to be analyzed in order of decreasing concentration. The computer performs calibration automatically. A linear fit should be used.

Calibration curves are to be printed for each run and included with the data package.

8.3 Instrument Start-Up

8.3.1 Turn on the analytical console, sampler, printer, and computer.

8.3.2 Turn on the nitrogen tank and verify that there is enough in the tank for the day's analysis and that the pressure is set at 30 psi.

8.3.3 When the DOS prompt appears, type HERC.

8.3.4 At the TRAACS main menu, select CHART AND RUN.

8.3.5 Press F4 B1. Should get "Error 129 from send cmd."

8.3.6 Press F4 CK. Should get 0.

8.3.7 Press DMO to electronically debubble.

8.3.8 Press F4 OP1 to start pumps.

8.3.9 Latch plattens and install air lines in the pinch valves.

8.3.10 Install the reagent lines in the appropriate reagent containers. Diagrams of the proper flow diagrams are attached. Allow reagents to flow for approximately 20 minutes until a good bubble pattern is obtained.

8.3.11 After the reagents have been flowing for at least 5 minutes, place the copper/cadmium reduction column in line on the nitrate channel.

Connect the inlet side first. The pumps should be turned off while doing this (press F4 QP1). Restart the pumps when finished installing the column (F4 OP1).

8.3.12 Press F2 to return to the main menu.

8.4 Automatic Base and Gain

8.4.1 Select BASE AND GAIN from the main menu.

8.4.2 Fill a cup with 3.0 mg/L standard and place in position 1 on the sampler.

8.4.3 Press F9 to turn on the recorder function of the printer.

8.4.4 At the "WHICH CHANNELS?" message, press 12 to activate the ammonia and nitrate channels. Use a chart speed of 60.

8.4.5 Press F7 to start the Base and Gain determination. It will terminate automatically.

8.4.6 Check the printed base and gain settings. If the gain is higher than normal, consult your supervisor.

8.4.7 Turn chart off (F9) and press F2 to return to the main menu.

8.5 Edit Run Program

8.5.1 Set up the sampler with the samples and standards. Standards should be in order of decreasing concentration starting at position 1 on the sampler.

Record the sample placed at each position on the bench sheet. As an example:

Cup 1	3.0 mg/L
Cup 2	2.0 mg/L
Cup 3	1.0 mg/L
Cup 4	0.5 mg/L
Cup 5	0.2 mg/L
Cup 6	0.1 mg/L
Cup 7	Blank (0.2% sulfuric acid)
Cup 8	LCS
Cup 9,10	Empty
Cup 11-20	10 Samples

8.5.2 Choose EDIT from the main menu.

8.5.3 Enter the File Name NO3NH3 and press F5 to read the file.

8.5.6 Verify that all the information displayed is correct. See the attached printout.

8.5.7 Press F3 or PGDN to go to the second screen of the editor.

8.5.8 The attached example is for a run of 10 samples. Additional samples can be added to the end of the tray protocol. This tray protocol would run the following samples:

P	Primer (Cup 1)
2N@7	2 Blanks (rinses) from cup 7
6C>1	6 Calibrants starting with cup 1
H@1	High Standard, carryover correction
2L@6	2 Low Standard, for carryover
N@7	Blank
I11@3	1 mg/L calibration verification
212@8	2 LCS's at from cup 8
N@7	Rinse
10S>11	10 samples starting at cup 11
I1@3	1 mg/L standard check
N@7	Rinse
E	End of run

For details on the tray protocol, see your supervisor or the instrument manual.

8.5.9 After entering the tray protocol, store the run program by pressing F6 twice.

8.5.10 Print the tray protocol by holding down the shift key and pressing PRT SC. Then press 0 and the screen will be printed.

8.5.11 Press F2 to return to the main menu.

8.6 Chart and Run

8.6.1 Choose CHART AND RUN from the main menu.

8.6.2 Press F9 to start recording. Use a chart speed of 60. The baseline should be about 5% scale and free of noise.

8.6.3 Press F7 to start the run. Enter the run program name (NO3NH3) and your initials or name at the appropriate prompts. You may also annotate the run with a comment.

8.6.4 Enter the file name for saving the data. The convention for filenames is YYMMDDX where YY is the year (e.g. 88), MM is the month (e.g. 01 for January, 11 for November) and DD is the day (e.g. 02, 30). X is a letter (A through Z) indicating the sequence in which different runs were made on a day.

8.6.5 The instrument will then automatically initiate the run and finally calculate and print the results.

8.6.6 Examine the results and rerun any off-scale samples at a dilution. All dilutions are to be made with 0.2% sulfuric acid. Also be alert to any other problems.

8.6.7 Press F2 to return to the main menu. If additional runs are to be made, edit the run program as above and restart.

8.7 Print Calibration Data

8.7.1 Select Retrieve from the main menu.

8.7.2 Press F6 and enter the file name for the stored data (chart file name).

8.7.3 Press F9 and enter the channel number for the calibration curve to be printed (1 for ammonia, 2 for nitrate). Press ENTER to display the calibration curve.

8.7.4 Hold in the SHIFT key and press PRT SC. Then press 1 to print the curve. Examine the curve for any problems, such as bad data points or nonlinearity. Print out calibration curves for both channels.

8.7.5 Press ENTER to return to the previous screen.

8.7.6 Press F8 and the equations for the calibration curves will be displayed. Print them by pressing (SHIFT) PRTSC 0.

8.7.7 Press F2 to return to the main menu.

8.8 Reprocessing Bad Data

If problems arise, it may be possible to correct them by reprocessing the data without rerunning the samples. See your supervisor.

8.9 Instrument Shut-Down

8.9.1 Disconnect both sides of the copper/cadmium reduction column and reconnect them to each other. Reconnect the transmission tubing to bypass the column. The pumps should be off while doing this. Disconnect the outlet side first.

8.9.2 Place all reagent lines in deionized water and rinse for 15 minutes.

8.9.3 Place all reagent lines in 5 N sulfuric acid and rinse for 15 minutes.

8.9.4 Place all reagent lines in deionized water and rinse for 15 minutes.

8.9.5 Press F4 QP1 to turn off the pumps.

8.9.6 Release the pump plattens.

8.9.7 Pull the air lines from the pinch valves and turn off the nitrogen.

8.9.8 Turn off the power to the computer, sampler, and analytical console.

8.10 Copper/Cadmium Column Cleaning Procedure

Perform this procedure only when a new column is installed or when the activation procedure fails to restore proper operation of the column.

8.10.1 Attach a syringe to the outlet of the column and draw through 1 ml 2 N nitric acid.

8.10.2 Draw 5 ml concentrated hydrochloric acid through the column followed by 5 ml deionized water. Repeat this step 3 more times.

8.10.3 Draw an additional 20 ml deionized water through the column, then activate as below.

8.11 Copper/Cadmium Column Activation Procedure

Perform this procedure when nitrate reduction drops by more than 10% or whenever the cleaning procedure has been used.

8.11.1 Follow the start-up procedure. After installing the column, place the buffer line in 2 N hydrochloric acid for 1 minute.

8.11.2 Place the buffer line in activating solution for 2 minutes, then in 2 N hydrochloric acid for 5 minutes.

8.11.3 Place the column back in line and allow 10 minutes equilibration time.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank (0.2% sulfuric acid) is required with every batch of 20 or less samples.

9.1.2 Two LCS samples are required with every batch of 20 lr less samples. Use an ERA nutrients sample.

9.1.3 Duplicates may be required for project specific QC.

9.1.4 Spikes may be required as project specific QC. Dilute 1 ml Intermediate Standard to 10 ml with sample. The spiking concentration is 1 mg/L.

9.1.5 A standard check is required after every 10 or less samples and at the end of each run.

9.2 Acceptance Criteria

9.2.1 Control limits for LCS samples are

	Accuracy	Precision
Ammonia (undistilled)	93 - 107%	10%
Ammonia (distilled)	80 - 120%	20%
Nitrate + Nitrite	91 - 109%	10%

9.2.2 No control limits for project specific QC have been established.

9.2.3 Standard checks must be within 10% of the expected value.

9.3 Corrective Action Required

9.3.1 Check for contamination in the reagents and standards. Be sure all reagents were made correctly and have not exceeded their expiration dates.

9.3.2 All samples and standards must contain 0.2% sulfuric acid.

9.3.3 Check system for obvious problems such as plugs and leaks.

9.3.4 Some samples may deactivate the copper/cadmium column, resulting in low standard checks for nitrate. Perform the activation procedure and reanalyze at a dilution.

9.3.5 If you are unable to locate and solve the problem, consult your supervisor.

10. Calculations

Concentrations are calculated automatically by the instrument. Record the calculated values on the bench sheet and multiply by any dilution factors. Review the printouts carefully to spot any problems.

If nitrate was required rather than nitrate + nitrite, subtract the nitrite value obtained by separate analysis from the nitrate + nitrite value obtained here.

11. Reporting

11.1 Reporting units are mg/L as N for water samples and mg/kg for soil samples. The units are the same for ammonia and nitrate + nitrite.

11.2 Results less than 0.1 mg/L are reported as ND. The detection limit must be raised if dilutions were made during sample prep or due to interferences.

11.3 Significant Figures

From the detection limit to 10 times the detection limit, report 1 significant figure.

Report higher concentrations with 2 significant figures.

Report LCS results with 3 significant figures.

11.4 LDMS Data Entry

The usual standards for data entry apply.

12. References

12.1 Method Source

Ammonia: EPA Method 350.1
Nitrate + Nitrite: EPA Method 353.2

12.2 Deviations from source method and rationale

The reagents and operating conditions were modified according to manufacturers instructions for the TRAACS system. The original method was written for the AAI and AAII.

The nitrate range was extended to 3 mg/l.

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-8
SOP Number: LM-RMA-1099
Title: Total Recoverable Phenolics

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

Section 9: Laboratory Control Samples are routinely used to access the precision and accuracy of analytical results, as detailed in QAPjP Appendix C-1.

Section 9.1.4: Duplicate analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.

Section 9.1.5: Matrix spike analysis will be performed at a frequency of one per batch of 20 prepared/analyzed samples.

Section 9.2: SW-846 methods, on which many Enseco methods are based, do not contain control limits for matrix spike and replicate analysis. The SW-846 methods do however contain limits on calibration checks. These checks are ±20 percent or greater, thus indicating that the accuracy limit on a matrix spike can never be greater than 80 to 120 percent. The 80 to 120 percent limits will be utilized.

Section 11: An example data reporting package is presented in QAPjP Appendix C-3.

STANDARD
OPERATING
PROCEDURE

Subject or Title: Total Recoverable Phenolics (Automated)		Page 1 of 10
SOP No.: LM-RMA-1099	Revision No.: Original	Effective Date: March 13, 1989
Supersedes: None		

1. Scope and Application

   1.1 This method measures steam-distillable phenolic materials which react with the color reagents under the conditions of the analysis.

   1.2 The detection limit is 0.01 mg/L as Phenol (1 mg/kg for nonaqueous matrices).

   1.3 This method is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes, and soil samples.

   1.4 The range extends from 0.01 mg/L to 0.5 mg/L (1 mg/kg to 40 mg/kg). The range can be extended by dilution of the samples.

   1.5 Approximate preparation time is 2 hours for a group of 10 samples. Analytical time is about 20 samples per hour.

2. Summary of Method

   The sample is acidified and distilled to separate phenolics from interfering compounds. Phenolics in the distillate react with 4-aminoantipyrene in the presence of potassium ferricyanide at pH 10 to form a reddish-brown dye, which is measured colorimetrically at 505 nm. This is the method commonly used at RMAL. A detection limit of 0.005 mg/L (1 mg/kg for nonaqueous matrices) can be achieved using SOP: LM-RMA-1048.

3. Comments

   3.1 Interferences

Prepared by: Lindsay Breyer	Date: February 27, 1989
Management Approval: [illegible] W. Pratt II	Date: 3/14/89
QA Officer Approval: [illegible] Redenbarger	Date: 3/15/89

3.1.1 Most direct interferences are eliminated by distillation of an acidified sample. Phenolic compounds distill with the water but interfering compounds do not.

3.1.2 Some phenolic compounds are not steam-distallable and will not be determined.

3.1.3 The colors produced by various phenolic compounds are not the same, so the response will depend on the compounds actually present in the samples. Phenol has been selected as the calibration standard since it is not possible to reproduce the mixture of compounds present in the samples. The result obtained will represent the minimum concentration of phenolics present in the sample.

3.1.4 Interference from sulfur compounds is eliminated by acidification and addition of copper sulfate.

3.1.5 Oxidizing agents such as chlorine will oxidize phenolic compounds and must be removed.

3.1.6 Oil may distill over and interfere with the analysis.

3.1.7 Aromatic amines may react with nitrite (if present) to produce phenolic compounds.

3.2 Helpful Hints
None.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco/RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.

4.2 Wear gloves and apron when handling concentrated acids, bases and solvents. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up any spill immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your supervisor or safety officer of any spills or exposures.

4.3 Wear gloves, apron and face shield when performing distillations. Distillations are to be performed under the slot hood.

4.4 Phenol is **extremely toxic** and can be absorbed through the skin. Handle only in a fume hood and wear gloves. In case of skin contact, flush with water for at least 15 minutes. Notify your supervisor or safety officer of any exposures.

4.5 Samples, reagents and other solutions containing high concentrations of toxic materials **must not** be flushed down the sinks, but are to be disposed of in suitable waste containers.

5. Samples Collection and Preservation

5.1 Samples are to be collected in glass containers and preserved by adding sulfuric acid to pH < 2 and refrigerating at 4RC.

5.2 The holding time is 28 days.

6. Apparatus

6.1 All-glass distillation apparatus consisting of 500 mL round-bottom flask with side arm, coil condenser, heating mantle with controller and associated adapters and hardware.

6.2 Recirculating chiller.

6.3 pH meter and electrode.

6.4 Technicon AutoAnalyzer II with sampler, pump, colorimeter, recorder and manifold shown in the attached diagram. Use 50 mm flow cells and 505 nm filters.

6.5 Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

7.1 Sulfuric Acid, 50%

Slowly add 50 mL concentrated sulfuric acid to 500 mL deionized water with constant mixing and cool. The reaction is very exothermic and should be done with extreme caution.

7.2 Boiling stones.

7.3 Copper Sulfate, 10%

Dissolve 100 g cupric sulfate 5-hydrate in deionized water and dilute to 1000 mL.

7.4 Ferrous Ammonium Sulfate Solution

Add 1 mL concentrated sulfuric acid to 500 mL deionized water. Add 1.1 g ferrous ammonium sulfate, mix until dissolved, and dilute to 1000 mL.

7.5 Sodium Hydroxide, 1N

Dissolve 40 g sodium hydroxide in deionized water, cool, and dilute to 1000 mL with deionized water. Store in a plastic bottle.

7.6 Buffered Potassium Ferricyanide

Dissolve 2.0 g potassium ferricyanide, 3.1 g boric acid and 3.75 g potassium chloride in 800 mL deionized water. Adjust the pH to 10.3 with 1 N sodium hydroxide and dilute to 1000 mL. Add 0.5 mL Brij-35 and mix. Prepare weekly.

7.7 4-Aminoantripyrene

Dissolve 0.65 g 4-aminoantipyrene in deionized water and dilute to 1000 mL.

7.8 Phenol Stock Standard, 1000 mg/L

Dissolve 1.000 g phenol in deionized water and dilute to 1000 mL.

7.9 Phenol Intermediate Standard, 10 mg/L

Dilute 1.0 mL 1000 mg/L Stock Standard to 100 mL with deionized water.

7.10 Working Standards

Dilute the 10 mg/L Intermediate Standard with deionized water as follows:

Aliquot (mL)	Final Vol. (mL)	Conc. (mg/L)
0	100	Blank
1.0	100	0.1
2.0	100	0.2
3.0	100	0.3
4.0	100	0.4
5.0	100	0.5

NOTE: The standards are not distilled with the samples.

7.11 pH test strips

7.12 Starch/iodide test strips

7.13 Lead Acetate test strips

8. Procedure

8.1 Sample Preparation

8.1.1 Measure and record the pH of all water samples. pH test strips may be used.

8.1.2 Check for residual chlorine with starch/iodide test strips. A blue to black color indicates a positive test. Record the result on the bench sheet.

8.1.3 Check for sulfide using lead acetate test strips. A dark color indicates the presence of sulfide. Record the result on the bench sheet.

8.1.4 Measure 200 mL sample into a distillation flask and add a few boiling stones. For soil and waste samples, use 2.0 g and add 200 mL deionized water. Be sure to adjust the pH of soil and waste samples before distillation. Record the exact weight on the bench sheet.

8.1.5 If the chlorine test was positive, add ferrous ammonium sulfate solution until a negative test is obtained.

8.1.6 If the pH is not < 2, add 50 % sulfuric acid dropwise until it is.

8.1.7 If the sulfide test was positive, add 2 mL 10% copper sulfate.

8.1.8 Assemble the distillation apparatus, turn on the cooling water and hood, and start the distillation. Capture the distillate in a 250 mL beaker.

8.1.9 When 150 to 175 mL distillate has been collected, turn off the heating mantle and allow to cool.

8.1.10 Add 25 to 30 mL deionzied water and resume distillation until 200 mL has been collected. Turn off the heating mantle and clean out the flask when cool. Do not overdistill the samples as this will lead to interferences in the analysis.

8.1.11 If the distillate is cloudy, filter through a prerinsed membrane filter.

8.1.12 Transfer the distillates to 250 mL glass bottles with teflon caps and refrigerate until they are analyzed.

8.1.13 At the end of the day, turn off the chiller.

8.2 Analysis

8.2.1 Set up the manifold shown in the attached diagram (Figure 1). Allow the colorimeter and recorder to warm up for at least 30 minutes while pumping reagents (pump deionized water through the sample line).

8.2.2 Continue pumping reagents until a steady baseline is obtained. If new pump tubing has been installed, up to 2 hours may be necessary. Adjust the baseline to the bottom of the charts with the baseline control.

8.2.3 Fill three cups with the 0.5 mg/L standard, place on the sampler and start. Use the standard cal control to adjust the peaks to full scale. Readjust the baseline if necessary.

8.2.4 Place the standards on the sampler in order of decreasing concentration. Follow these with the prep blank, the DCSs and the samples.

8.2.5 Start the sampler. Dilute and reanalyze any samples reading off-scale. Be sure to analyze a blank and standard check after every 10 or less samples and again at the end of the run.

8.2.6 At the end of the day, flush the system thoroughly with deionized water and turn off the instrument.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank (deionized water) is required with every batch of 20 or less samples. The blank must be taken through the entire prep and analysis with the samples.

9.1.2 Two DCS samples are required with each batch of 20 or less samples. Use an ERA cyanide/phenol sample. These must be taken through the entire prep and analysis with the samples.

9.1.3 A blank and standard check are required after every 10 or less samples.

9.1.4 Duplicates may be required for project specific QC.

9.1.5 Spikes may be required for project specific QC. Add 5.0 mL of the 10 mg/L intermediate standard to 200 mL sample and distill. The spiking concentration is 0.25 mg/L (25 mg/kg).

9.2 Acceptance Criteria

9.2.1 If the blank reads greater than 3 times the detection limit, consult your supervisor.

9.2.2 DCS recovery must be 88 - 122%.

9.2.3 The RPD for DCS samples must be less than 20%.

9.2.4 Standard checks must be within 20% of the expected value.

9.2.5 There are no acceptance criteria for project specific QC.

9.2.6 The calibration curve must have a correlation coefficient of at least 0.995.

9.3 Corrective Action Required

9.3.1 Negative peaks usually indicate that the sample pH is too low. Add 1 N sodium hydroxide dropwise to adjust the pH to between 5 and 6.

9.3.2 Follow the corrective action procedures outlined in the current Enseco QA Plan. If problems persist, consult your supervisor.

10. Calculations

10.1 Measure and record the peak heights of all samples and standards in the order in which they were analyzed.

10.2 Use the data for the standards to determine a calibration curve using a linear least squares program.

10.3 Calculate the sample concentrations from their peak heights. Multiply by any dilutions made during prep and/or analysis.

11. Reporting

11.1 Reporting units are mg/L for water samples and mg/kg for soil samples.

11.2 Samples less than the 0.01 mg/L (1 mg/kg) are reported as N.D.

11.3 Significant Figures:

Range	Water Samples	Soils/Wastes
0.01 - 0.09	1	----
0.10 - 0.99	2	----
1.0 - 9.9	2	1
10 - 99	2	2
100+	3	3

11.4 LDMS Data Entry

11.4.1 The usual standards for data entry apply.

12. References

12.1 Method source: EPA Methods 420.1, 420.2

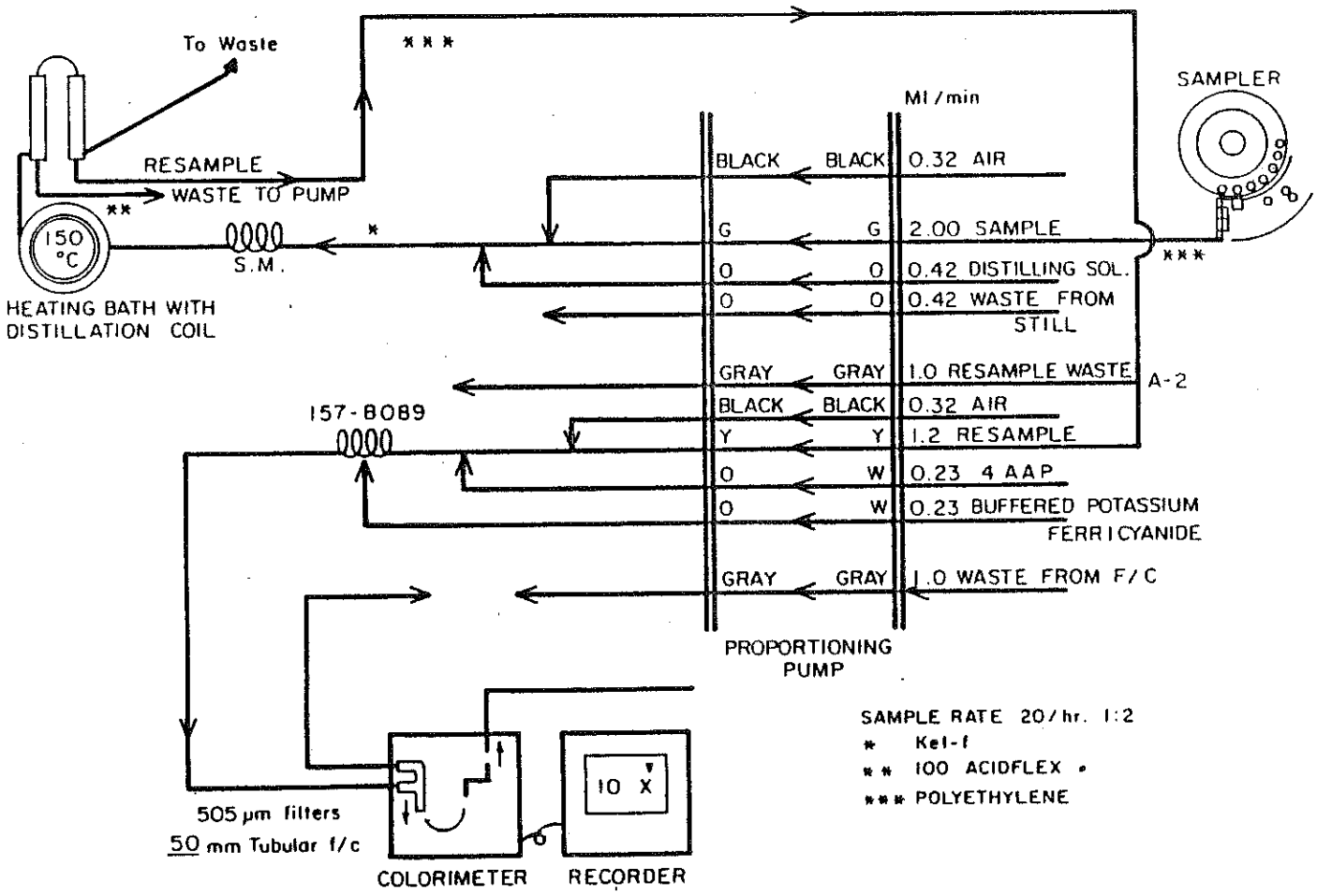
12.2 Deviations from source method and rationale.

12.2.1 The two source methods were combined to allow manual distillation and automated colorimetric analysis.

12.2.2 There is a discrepancy between the preservation methods and holding times given in the method and those given in the table of containers and preservatives at the front of the methods book. We have chosen to use sulfuric acid to adjust the sample pH to 2 since this has been done traditionally at RMAL.

12.2.3 The size of the distillation apparatus and volumes of sample and reagent were reduced to conserve space and speed up the analysis.

Figure 1. Automated Phenol Analysis Schematic



ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-9
SOP Number: LM-RMA-4007
Title: Phenoxyacid Herbicides
by Electron Capture Gas Chromatography

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

A new SOP version is presented with this QAPjP submittal (Revision 1).

Section 1.1: Target compounds are listed in this section. Parameter retention time is variable with instrument condition and may vary slightly from calibration to calibration. Retention times recorded for an actual calibration are found in QAPjP Appendix B-9A.

Section 1.3: Linear dynamic range of compounds is determined from the instrument calibration, and normally covers one order of magnitude of analyte concentration. See QAPjP Appendix B-9A for an example of a five point instrument calibration. The standard concentrations used for various analytes are listed as the "true" value, in ug/mL.

Section 6.2: For the identification of a compound, Enseco requires mutual agreement that there is a detectable value on each column. The lower value from the two columns (above the reporting limit) is reported.

Section 13.: See QAPjP Appendix C-3 for an example of the data reporting package.

STANDARD
OPERATING
PROCEDURE

Subject or Title:

Analysis of Environmental Samples for Chlorinated Phenoxyacid Herbicides (Modified EPA Method 615/8150)

SOP No.:	Revision No.:	Effective Date:
LM-RMA-4007	3.0	December 10, 1991

Supersedes: Revision 2.0

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1. Scope and Application

   1.1 Analytes

Prepared by:	Date:
GC Department	December 6, 1991
Management Approval: Debra Henderson	Date: 12-31-91
QA Officer Approval: Gary Toy	Date: 1-8-92

Parameter	STORET No.	CAS No.
2,4-D	39736	94-75-7
Dalapon		75-99-0
2,4-DB		94-82-6
Dicamba		1918-00-9
Dichlorprop		120-36-5
Dinoseb		88-85-7
MCPA		94-74-6
MCPP		93-65-2
2,4,5-T	39740	93-76-5
2,4,5-TP (Silvex)	39760	93-72-1
DCAA		19719-28-9

1.2 Matrices

This method is applicable to the analysis of chlorinated phenoxyacid herbicides in water, soil, solid, leachate, and vegetation samples.

1.3 Linear Range of Instrument

Calibration standards are run at 6 levels and a quadratic calibration curve is generated for each compound. The calibration range normally covers one order of magnitude.

1.4 Analysis time is approximately 30 minutes per sample or standard.

1.5 Sample preparation requires at least three days for completion.

2. Summary of Method

2.1 This method is applicable to the chlorinated phenoxyacid herbicides listed in 1.1, their salts, and esters. The herbicide is hydrolyzed to the acid form, solvent extracted, then converted to the methyl ester and analyzed by electron-capture gas chromatography.

3. Comments

3.1 Helpful Hints

After analyzing herbicides it is necessary to change the liner and cut the front ends of the columns every seven days. After performing maintenance, raise injection port temperature to 260°C, oven to 280°C, and detector to 350°C for a few hours while injecting hexane every two minutes. This procedure effectively cleans the GC system.

4. Safety Issues

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis.

5. Sample Collection, Preservation and Handling

5.1 Grab samples must be collected in glass containers. Conventional sampling practices should be followed; however, the bottle must not be pre-rinsed with sample before collection.

5.2 The samples must be iced or refrigerated at 4°C from the time of collection until extraction.

5.3 All water samples must be extracted within 7 days and all soils within 14 days of collection. All samples must be analyzed within 40 days from the date that extraction begins.

6. Apparatus and Materials

6.1 Balance - Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2 Gas chromatograph - Analytical system complete with gas chromatograph suitable for split/splitless capillary injection and all required accessories including: syringes, analytical columns, gases, detector and a data system or strip-chart recorder to measure peak areas. Enseco-RMAL uses a VG data system to quantify data.

6.2.1 Column 1 - RT_X-5, 30 m, 0.25 mm ID, 0.25 um film thickness or equivalent.

6.2.2 Column 2 - RT_X-20, 30 m, 0.25 mm ID, 0.25 um film thickness or equivalent.

6.2.3 A guard column - 5 m, 0.25 mm with no coating.

6.2.4 A glass "Y" connector - connects both analytical columns to the guard column.

6.2.5 Detectors - electron capture.

7. Reagents and Standards

7.1 Hexane - Pesticide quality or equivalent.

7.2 Calibration standard solutions.

7.2.1 The methyl ester standards used to calibrate the GC system are purchased as a certified solution. Serial dilutions are made to obtain standard solutions at six different levels.

7.2.2 Solutions used by the prep lab for spikes and methylation check standards are prepared from pure standard materials. These standards are all in the acid form and at a concentration of approximately 1.0 ug/uL.

7.2.2.1 Prepare primary standard solutions by accurately weighing about 0.0100 g of pure acids. Dissolve the material in pesticide quality hexane and dilute to volume in a 10 mL volumetric flask. Larger volumes can be used at the convenience of the analyst. If compound purity is certified at 99% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

8. Procedure

8.1 Method Summary

This method provides extraction and esterification procedures for the analysis of chlorinated phenoxyacid herbicides. The esters are hydrolyzed with potassium hydroxide, and extraneous organic material is removed by a solvent wash. After acidification, the acids are extracted with solvent and converted to their methyl esters using diazomethane as the derivitizing agent. After excess reagent is removed, the esters are analyzed by gas chromatography employing an electron capture detector (Goerlitz and Lamar, 1967).

8.2 Sample Preparation and Extraction - See RMAL SOP No. LM-RMA-4044

9. Analysis

9.1 Sample analyses are performed simultaneously on dissimilar analytical columns.

9.1.1 The linear velocity ($\bar{u}$) should be approximately 40 cm/second. A typical temperature program for this analysis is as follows.

T_1	45°C
t_1	1.0 minute
R_1	20 °C/minute
T_2	160°C
t_2	4.0 minutes
R_2	1.3 °C/minute
T_3	180°C
t_3	0.0 minute
R_3	50.0 °C/minute
T_4	280°C
t_4	1.0 minute

Recommended GC conditions are as follows:

injector 240°C
detector 325°C
purge on 0.60 minute
carrier gas H_2
auxiliary gas N_2
volume injected 1-2 uL

9.2 Calibration of Instrument

9.2.1 Run six levels of standards to generate a 6-point quadratic calibration curve for each standard. RSE values less than 15% are required to quantify data. A mid-level calibration check standard is run after every 10 samples and at the end of the run. Insert a hexane blank before each check standard. If the check standards are within 20% difference from the curve the instrument is in control and data may be quantified. If the change in response factors exceeds 20% for selected compounds, consult an experienced analyst. The recommended internal standard, 4,4'-dibromooctafluorobiphenyl, is added to all samples and standards at 0.025 ug/mL. Samples may be quantified using internal standard calculations if the percent difference between the areas of the internal standard in the sample and the standard is $\leq$ 30%.

10. QA/QC Requirements

10.1 QC samples: With each set of samples methylated, a Methylation Standard is run consisting of 2,4-D, Silvex, and 2,4-DB to determine if the methylation step is working properly. There are no recovery limits established for the Methylation Standard. Recoveries of greater than or equal to 80% are desirable.

10.2 For standard product herbicide tests two lab control standards are prepared for every twenty samples and a spike control standard is prepped with every batch. Recoveries for both soils and waters are based on historical data and are periodically updated. The relative percent difference of the two replicates is limited to 20%. 80% of all QC values are required to be within limits before proceeding. A reagent blank is extracted with every group of samples.

10.3 DCAA is used as the surrogate.

10.4 Corrective action: See SOP LP-RMA-0022; Enseco QAPP, Revision 3.4, April 1991, and SOP M-EQA-0002.

11. Calculations: ug/mL can be read directly from the internal or external standard table.

11.1 For waters:

$$\frac{ug}{mL} \text{ of analyte} \times \frac{\text{final vol (mL)}}{\text{liters extracted (L)}} \times \text{dilution factor} = \frac{ug}{L}$$

11.2 For soils:

$$\frac{ug}{mL} \text{ of analyte} \times \frac{\text{final vol (mL)}}{\text{amount extracted (kg)}} \times \text{dilution factor} = \frac{ug}{kg}$$

Reporting units are ug/L for liquids and ug/kg for solids.

11.3 Unless specifically requested by the client, two significant figures are reported. Values below detection limit are reported as not detected (ND).

12. References

12.1 "Method for Chlorinated Phenoxy Acid Herbicides in Industrial Effluents" Federal Register, Vol. 38, No. 75, Pt II.

12.1.1 "Simultaneous Determination of 2,4-D, 2,4,5-T and 2-Methoxy-3,6-Dichlorobenzoic Acid in Soil and Water by GC/ECD" by R. Purkazastha, J. Agric and Food Chem., 74, 22, 3, p. 453-458.

12.2 USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460, November, 1986, SW-846, Third Edition, Method 8150.

13. Reporting Limits

The following reporting limits have been established based upon MDL data and the linear range of the detector.

Target Compound	Water (ug/L)	Soil (mg/kg)	Waste (mg/kg)
Dalapon	5.0	100	0.10
Dicamba	0.50	10	0.010
MCPP	250	5000	5.0
MCPA	250	5000	5.0
Dichlorprop	1.0	20	0.020
2,4-D	2.0	40	0.040
2,4,5-TP (Silvex)	0.50	10	0.010
2,4,5-T	0.50	10	0.010
2,4-DB	5.0	100	0.10
Dinoseb	1.0	20	0.020

Thu May 7 22:19:20 1992 Page 1

Reduced by: L.L Date: 5/7/92
Reviewed by: _____ Date: _____

Initial Calibration Report

Project: HRB05_1, Channel: 63, Analysis: N07MAY92, Node: DENCR3
Analyst: SMCCOOL, Calibration File: N0507, MCRPT Method: HRBA

Calibration curve type: Linear NOT forced through zero

Sample	Level ID	Description
3	L5	HRB CSA 20% T=ST #5897
4	L4	HRB CSA 10% T=ST #5897
5	L3	HRB CSA 5% T=ST #5897
6	L2	HRB CSA 4% T=ST #5897
7	L1	HRB CSA 2% T=ST #5897

%RSD Measured	%RSD Maximum	Compound
7.0	20.0	Dalapon
9.8	20.0	DCAA
7.0	20.0	Dicamba
6.5	20.0	MCPP
7.8	20.0	MCPA
8.9	20.0	Dichlorprop
9.0	20.0	2,4-D
3.2	20.0	Silvex
3.3	20.0	2,4,5-t
6.6	20.0	2,4-DB
6.6	20.0	Dinoseb

all RSD's < 10%

	Samp 3 Meas True %Diff	Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Samp 7 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
Dalapon								
	0.9462	0.4869	0.2627	0.2155	0.09515			
	1.000	0.5000	0.2500	0.2000	0.1000			
	-5.4	-2.6	5.1	7.8	-4.9			
DCAA								
	0.4562	0.2455	0.1328	0.1111	0.04658			
	0.5000	0.2500	0.1250	0.1000	0.05000			
	-8.8	-1.8	6.3	11.1	-6.8			
Dicamba								
	0.09421	0.04922	0.02592	0.02170	0.009517			
	0.1000	0.05000	0.02500	0.02000	0.01000			
	-5.8	-1.6	3.7	8.5	-4.8			
MCPP								
	95.68	48.55	25.86	21.66	9.549			
	100.0	50.00	25.00	20.00	10.00			
	-4.3	-2.9	3.5	8.3	-4.5			
MCPA								
	94.42	48.38	26.19	21.90	9.455			
	100.0	50.00	25.00	20.00	10.00			
	-5.6	-3.2	4.7	9.5	-5.5			
Dichlorprop								
	0.2309	0.1230	0.06542	0.05537	0.02346			
	0.2500	0.1250	0.06250	0.05000	0.02500			
	-7.6	-1.6	4.7	10.7	-6.2			
2,4-D								
	0.4599	0.2459	0.1324	0.1100	0.04689			
	0.5000	0.2500	0.1250	0.1000	0.05000			
	-8.0	-1.7	5.9	10.0	-6.2			
Silvex								
	0.09866	0.04898	0.02524	0.02090	0.009790			
	0.1000	0.05000	0.02500	0.02000	0.01000			
	-1.3	-2.0	1.0	4.5	-2.1			
2,4,5-t								
	0.09906	0.04934	0.02473	0.02102	0.009824			
	0.1000	0.05000	0.02500	0.02000	0.01000			
	-0.9	-1.3	-1.1	5.1	-1.8			
2,4-DB								
	2.377	1.224	0.6437	0.5429	0.2385			
	2.500	1.250	0.6250	0.5000	0.2500			
	-4.9	-2.1	3.0	8.6	-4.6			

Samp 3	Samp 4	Samp 5	Samp 6	Samp 7			
Meas	Meas	Meas	Meas	Meas	Meas	Meas	Meas
'rue	True	True	True	True	True	True	True
%Diff	%Diff	%Diff	%Diff	%Diff	%Diff	%Diff	%Diff
Dinoseb							
2.377	1.224	0.6437	0.5429	0.2385			
2.500	1.250	0.6250	0.5000	0.2500			
-4.9	-2.1	3.0	8.6	-4.6			

Thu May 7 22:19:44 1992 Page 1

Reduced by: L-L Date: 5/7/92 Initial Calibration Report
Reviewed by: _____ Date: _____

Project: HRB05_1, Channel: 64, Analysis: N07MAY92, Node: DENCR3
Analyst: SMCCOOL, Calibration File: N0507, MCRPT Method: HRBA

Calibration curve type: Linear NOT forced through zero

Sample	Level ID	Description
3	L5	HRB CSA 20% T=ST #5897
4	L4	HRB CSA 10% T=ST #5897
5	L3	HRB CSA 5% T=ST #5897
6	L2	HRB CSA 4% T=ST #5897
7	L1	HRB CSA 2% T=ST #5897

%RSD Measured	Maximum	Compound
6.3	20.0	Dalapon
11.7	20.0	DCAA
13.9	20.0	Dicamba
7.7	20.0	MCPP
15.1	20.0	MCPA
9.7	20.0	Dichlorprop
8.0	20.0	2,4-D
5.7	20.0	Silvex
5.3	20.0	2,4,5-T
7.8	20.0	Dinoseb
8.1	20.0	2,4-DB

all RSD's < 16%

	Samp 3 Meas True %Diff	Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Samp 7 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
Dalapon								
	0.9427	0.4934	0.2637	0.2117	0.09571			
	1.000	0.5000	0.2500	0.2000	0.1000			
	-5.7	-1.3	5.5	5.9	-4.3			
DCAA								
	0.4456	0.2464	0.1341	0.1131	0.04594			
	0.5000	0.2500	0.1250	0.1000	0.05000			
	-10.9	-1.4	7.3	13.1	-8.1			
Dicamba								
	0.08979	0.04919	0.02523	0.02397	0.009105			
	0.1000	0.05000	0.02500	0.02000	0.01000			
	-10.2	-1.6	0.9	19.8	-8.9			
MCPP								
	95.24	47.76	26.56	21.65	9.475			
	100.0	50.00	25.00	20.00	10.00			
	-4.8	-4.5	6.2	8.3	-5.3			
MCPA								
	88.41	47.15	27.67	23.44	8.944			
	100.0	50.00	25.00	20.00	10.00			
	-11.6	-5.7	10.7	17.2	-10.6			
Dichlorprop								
	0.2196	0.1318	0.06584	0.05336	0.02366			
	0.2500	0.1250	0.06250	0.05000	0.02500			
	-12.1	5.5	5.3	6.7	-5.4			
2,4-D								
	0.4644	0.2450	0.1334	0.1080	0.04722			
	0.5000	0.2500	0.1250	0.1000	0.05000			
	-7.1	-2.0	6.7	8.0	-5.6			
Silvex								
	0.09531	0.04913	0.02608	0.02121	0.009606			
	0.1000	0.05000	0.02500	0.02000	0.01000			
	-4.7	-1.7	4.3	6.1	-3.9			
2,4,5-T								
	0.09613	0.04960	0.02517	0.02149	0.009655			
	0.1000	0.05000	0.02500	0.02000	0.01000			
	-3.9	-0.8	0.7	7.5	-3.4			
Dinoseb								
	0.2339	0.1246	0.06326	0.05536	0.02372			
	0.2500	0.1250	0.06250	0.05000	0.02500			
	-6.4	-0.4	1.2	10.7	-5.1			

Samp 3	Samp 4	Samp 5	Samp 6	Samp 7			
Meas	Meas	Meas	Meas	Meas	Meas	Meas	Meas
True	True	True	True	True	True	True	True
%Diff	%Diff	%Diff	%Diff	%Diff	%Diff	%Diff	%Diff
2,4-DB							
0.9342	0.4951	0.2553	0.2218	0.09457			
1.000	0.5000	0.2500	0.2000	0.1000			
-6.6	-1.0	2.1	10.9	-5.4			

Injection Report

Acquired on 7-MAY-1992 at 15:37

RMAL Chromatography System on DENCR3

Analyst Name : SMCCOOL
Lims Id : 2uL INJECTED
Comment : HP5890 Dual ECD Columns/RTx-5,RTx-20,0.25um,30m,0.25mm id
Method Title : METHOD FOR HERBICIDES
Sample Name : HRB CSA 20% T=ST #5897
Sample Id : VER # 5897
Sample Type : Standard Amount=1.00000
Bottle No : 3

L.L 5/7/92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
3	3.484	3.493	301801	604957	0.94619	Dalapon	2.1
10	9.520	9.538	193226	424650	0.45625	DCAA	2.1
11	9.738	9.751	131953	281089	0.09421	Dicamba	2.4
12	10.156	10.142	111434	307053	95.67641	MCPP	2.9
13	10.413	10.396	142940	456798	94.42266	MCPA	3.2
14	11.169	11.182	108076	311341	0.23089	Dichlorprop	2.9
15	11.578	11.604	164973	681266	0.45994	2,4-D	4.0
18	12.533	12.547	88567	240917		D-BOB	2.9
21	14.316	14.338	147223	632280	0.09866	Silvex	4.0
22	15.120	15.164	118425	649501	0.09906	2,4,5-t	4.5
C1		17.210	274770	1621867	2.37675	2,4-DB/Dinoseb	
24	17.142	17.210	107202	534531	0.75112	- C1	5.3A
25	17.253	17.210	167568	1087336	1.57760	- C1	5.6

Totals			
Unknowns	98548	288798	N/A
Quantified	1783389	6211718	194.86102
Grand Total	1881937	6500516	194.86102

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

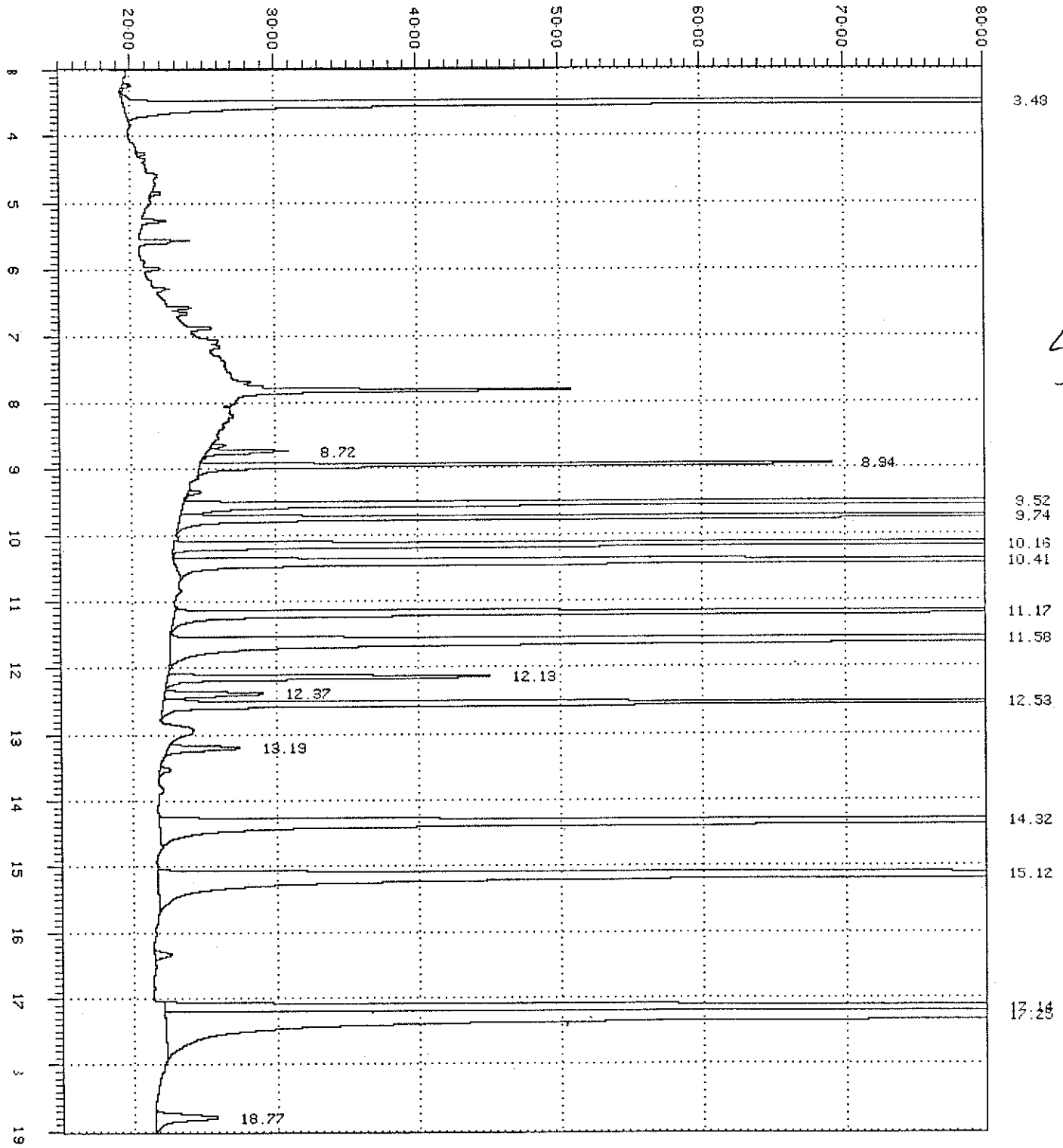
Method..... HERBUS1
Run sequence..... N0507
Calibration..... N0507
Internal standard calibration using area
Calibration last modified on 7-MAY-1992 at 22:18

Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[HRB05_1] 63 N07MAY92,3,1

HRB CSA 20% T=ST #5897. Amount : 1.000.
Inst.N/HP5890-Dual ECD Columns/RTx-5 .25um,30m,.25mm
Acquired on 7-MAY-1992 at 15:37
Reported on 7-MAY-1992 at 22:20
Box 1 (of 1)



L.C
5/7/92

Injection Report

Acquired on 7-MAY-1992 at 15:37

RMAL Chromatography System on DENCR3

Analyst Name : SMCCOOL
Lims Id : 2uL INJECTED
Comment : HP5890 Dual ECD Columns/RTx-5,RTx-20,0.25um,30m,0.25mm id
Method Title : METHOD FOR HERBICIDES
Sample Name : HRB CSA 20% T=ST #5897
Sample Id : VER # 5897
Sample Type : Standard Amount=1.00000
Bottle No : 3

L.L
5/7/92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
3	4.040	4.044	463472	1045627	0.94267	Dalapon	1.9
5	11.133	11.138	273019	778992	0.44558	DCAA	2.7
6	11.547	11.551	191903	530844	0.08979	Dicamba	2.7
7	11.827	11.804	179698	552778	95.24388	MCPP	2.9
8	12.502	12.471	215859	803932	88.41178	MCPA	3.7
11	13.440	13.449	152544	559131	0.21964	Dichlorprop	3.5
12	13.902	13.907	138083	457850		D-BOB	3.2
13	14.489	14.502	246823	1278651	0.46437	2,4-D	4.3
19	17.738	17.756	211139	1114502	0.09531	Silvex	5.1
21	19.556	19.587	170892	1139165	0.09613	2,4,5-T	5.6
23	21.684	21.702	216416	1460740	0.23388	Dinoseb	6.1
24	22.111	22.147	168140	1292510	0.93424	2,4-DB	6.7

Totals			
Unknowns	45637	239079	N/A
Quantified	2627985	11014720	187.17728
Grand Total	2673623	11253799	187.17728

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

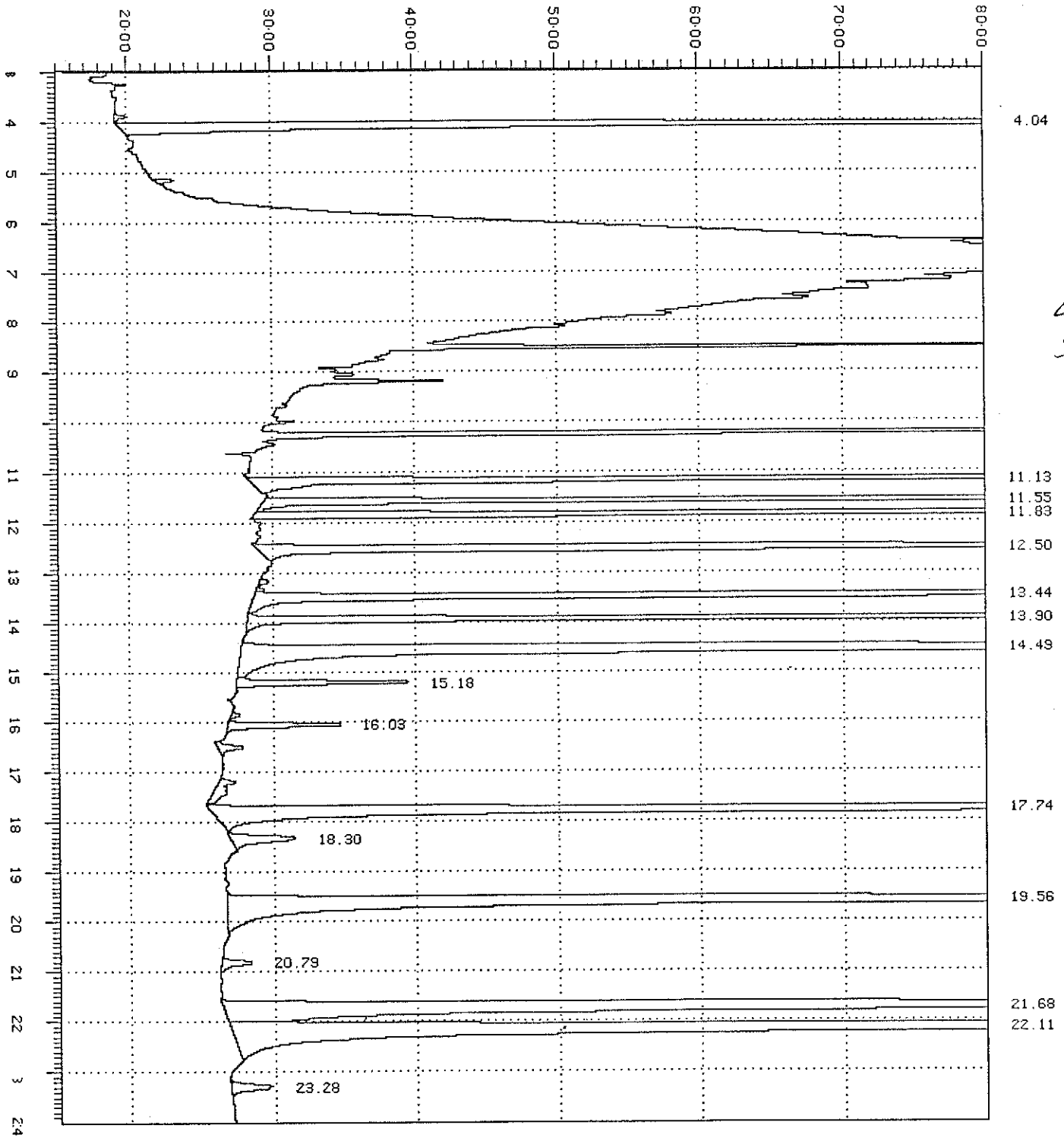
Method..... HERBUS1
Run sequence..... N0507
Calibration..... N0507
Internal standard calibration using area
Calibration last modified on 7-MAY-1992 at 22:17

Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[HRB05_1] 64 N07MAY92,3,1

HRB CSA 20% T=ST #5897. Amount : 1.000.
Inst.N/HP5890-Dual ECD Columns/RTx-5 .25um,30m,.25mm
Acquired on 7-MAY-1992 at 15:37
Reported on 7-MAY-1992 at 22:22
Box 1 (of 1)



L.L
5/7/92

Injection Report

Acquired on 7-MAY-1992 at 16:15

RMAL Chromatography System on DENCR3

Analyst Name : SMCCOOL
Lims Id : 2uL INJECTED
Comment : HP5890 Dual ECD Columns/RTx-5,RTx-20,0.25um,30m,0.25mm id
Method Title : METHOD FOR HERBICIDES
Sample Name : HRB CSA 10% T=ST #5897
Sample Id : VER # 5897
Sample Type : Standard Amount=1.00000
Bottle No : 4

L.L. 5/7/92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
2	3.493	3.493	165315	319057	0.48695	Dalapon	2.1A
10	9.533	9.538	99896	236978	0.24546	DCAA	2.4
11	9.751	9.751	66575	148877	0.04922	Dicamba	2.4
12	10.147	10.142	65393	158963	48.54819	MCPP	2.7
13	10.400	10.396	87513	249804	48.38334	MCPA	2.7
14	11.178	11.182	56581	169885	0.12298	Dichlorprop	2.9
15	11.596	11.604	79629	374915	0.24586	2,4-D	4.3
18	12.547	12.547	84562	238370		D-BOB	2.9
20	14.333	14.338	65759	311475	0.04898	Silvex	4.3
21	15.151	15.164	49787	319655	0.04934	2,4,5-t	5.1
C1		17.210	130574	842027	1.22430	2,4-DB/Dinoseb	
22	17.173	17.210	47817	230110	0.29967	- C1	5.3A
23	17.276	17.210	82757	611916	0.87659	- C1	6.7

Totals			
Unknowns	51279	157628	N/A
Quantified	951585	3370006	99.40462
Grand Total	1002863	3527635	99.40462

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

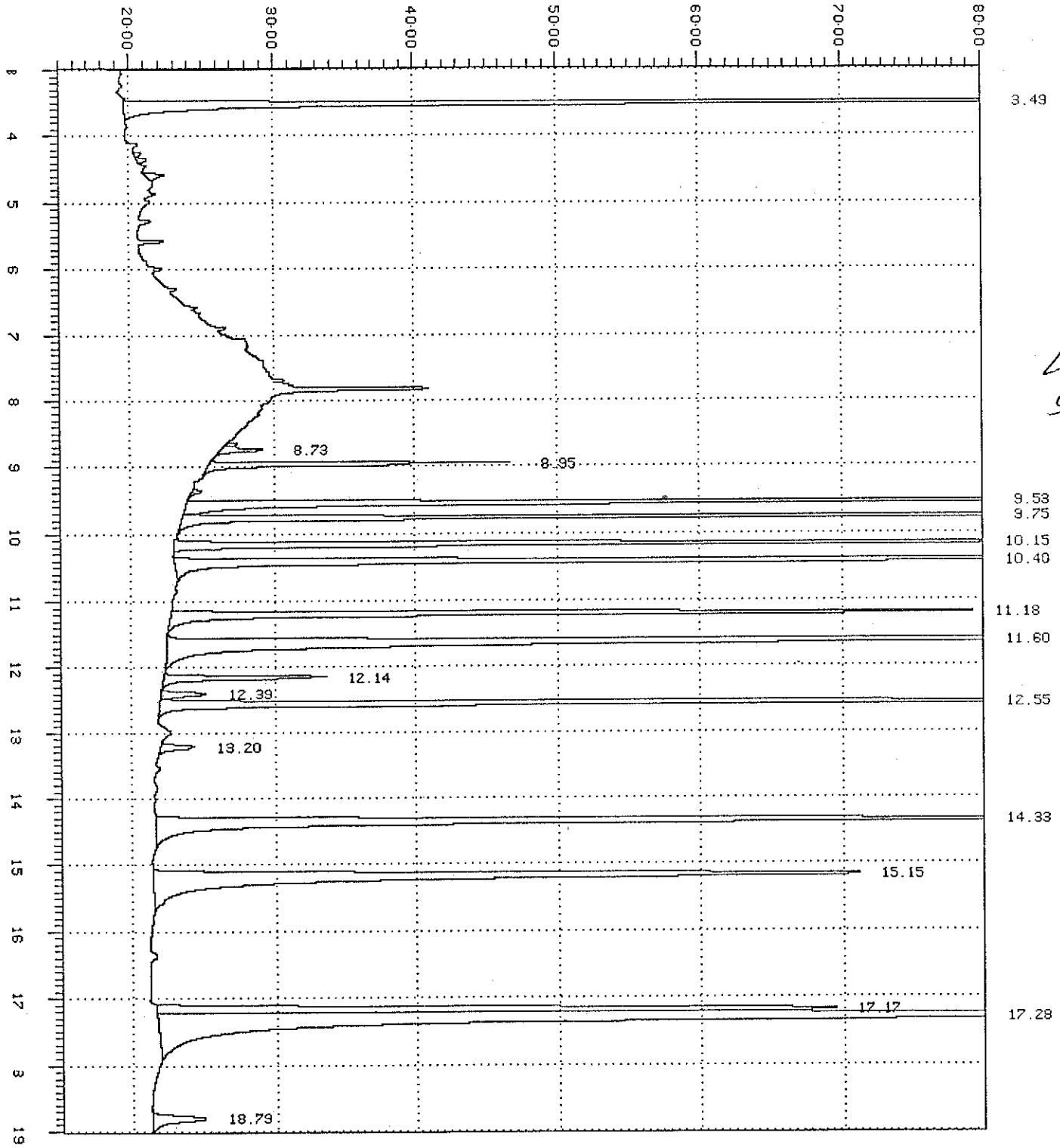
Method..... HERBUS1
Run sequence..... N0507
Calibration..... N0507
Internal standard calibration using area
Calibration last modified on 7-MAY-1992 at 22:18

Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[HRB05_1] 63 N07MAY92,4,1

HRB CSA 10% T=ST #5897. Amount : 1.000.
Inst.N/HP5890-Dual ECD Columns/RTx-5 .25um,30m,.25mm
Acquired on 7-MAY-1992 at 16:15
Reported on 7-MAY-1992 at 22:20
Box 1 (of 1)



L.L.
5/7/92

Injection Report

Acquired on 7-MAY-1992 at 16:15

RMAL Chromatography System on DENCR3

Analyst Name : SMCCOOL
Lims Id : 2uL INJECTED
Comment : HP5890 Dual ECD Columns/RTx-5,RTx-20,0.25um,30m,0.25mm id
Method Title : METHOD FOR HERBICIDES
Sample Name : HRB CSA 10% T=ST #5897
Sample Id : VER # 5897
Sample Type : Standard Amount=1.00000
Bottle No : 4

L.L 5/7/92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
3	4.049	4.044	258059	569786	0.49337	Dalapon	1.9
7	11.142	11.138	147118	447327	0.24640	DCAA	2.9
8	11.556	11.551	103199	294127	0.04919	Dicamba	2.9
9	11.818	11.804	100490	260338	47.75597	MCPP	2.7
10	12.489	12.471	126933	409047	47.14702	MCPA	3.5
11	13.453	13.449	83300	340468	0.13181	Dichlorprop	3.5
12	13.916	13.907	135152	456231		D-BOB	3.2
13	14.507	14.502	125161	697742	0.24502	2,4-D	5.1
18	17.760	17.756	105422	585212	0.04913	Silvex	5.1
20	19.582	19.587	81702	589691	0.04960	2,4,5-T	6.1
22	21.702	21.702	113019	794802	0.12455	Dinoseb	6.1
23	22.138	22.147	81857	699370	0.49506	2,4-DB	6.9

Totals			
Unknowns	31132	175548	N/A
Quantified	1461411	6144140	96.78711
Grand Total	1492544	6319688	96.78711

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

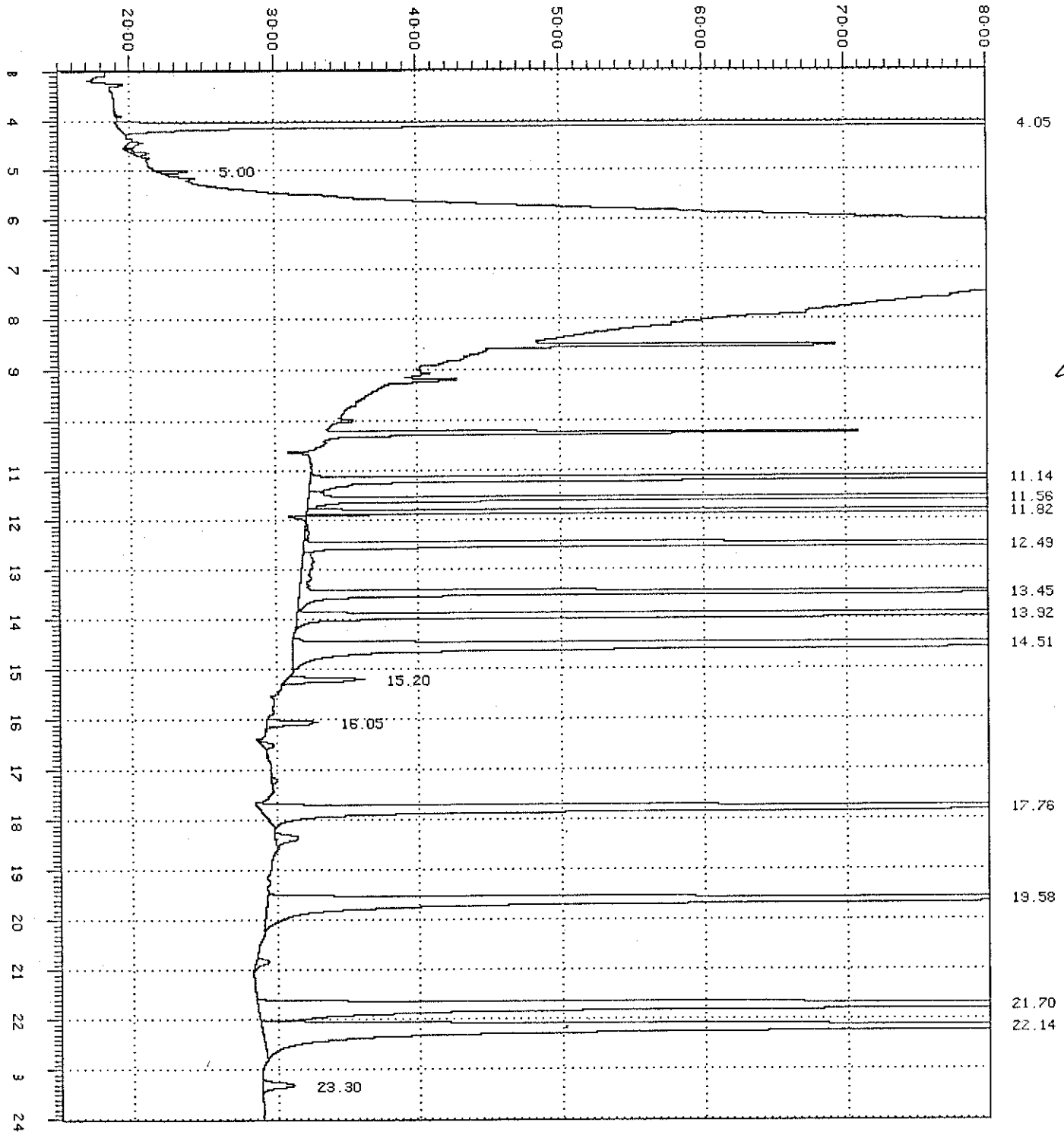
Method..... HERBUS1
Run sequence..... N0507
Calibration..... N0507
Internal standard calibration using area
Calibration last modified on 7-MAY-1992 at 22:17

Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[HRB05_1] 64 N07MAY92,4,1

HRB CSA 10% T=ST #5897. Amount : 1.000.
Inst.N/HP5890-Dual ECD Columns/RTx-5 .25um,30m,.25mm
Acquired on 7-MAY-1992 at 16:15
Reported on 7-MAY-1992 at 22:22
Box 1 (of 1)



L.C.
5/7/92

Injection Report

Acquired on 7-MAY-1992 at 16:53

RMAL Chromatography System on DENCR3

Analyst Name : SMCCOOL
Lims Id : 2uL INJECTED
Comment : HP5890 Dual ECD Columns/RTx-5,RTx-20,0.25um,30m,0.25mm id
Method Title : METHOD FOR HERBICIDES
Sample Name : HRB CSA 5% T=ST #5897
Sample Id : VER # 5897
Sample Type : Standard Amount=1.00000
Bottle No : 5

L.C 5/7/92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
2	3.493	3.493	89036	190158	0.26271	Dalapon	2.1
12	9.538	9.538	55920	144864	0.13283	DCAA	2.4
13	9.751	9.751	36597	85338	0.02592	Dicamba	2.4
14	10.142	10.142	39513	92949	25.86383	MCPP	2.7
15	10.396	10.396	53489	158660	26.18706	MCPA	2.9
16	11.182	11.182	32398	100191	0.06542	Dichlorprop	3.2
17	11.604	11.604	42759	225297	0.13237	2,4-D	4.5
20	12.547	12.547	87672	248262		D-BOB	2.9
22	14.338	14.338	33009	168036	0.02524	Silvex	4.5
23	15.164	15.164	24567	166427	0.02473	2,4,5-t	5.9
C1		17.210	45620	476751	0.64365	2,4-DB/Dinoseb	
24	17.284	17.210	45620	476751	0.64365	- C1	10.4

Totals			
Unknowns	34994	117712	N/A
Quantified	540581	2056934	53.36377
Grand Total	575575	2174646	53.36377

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

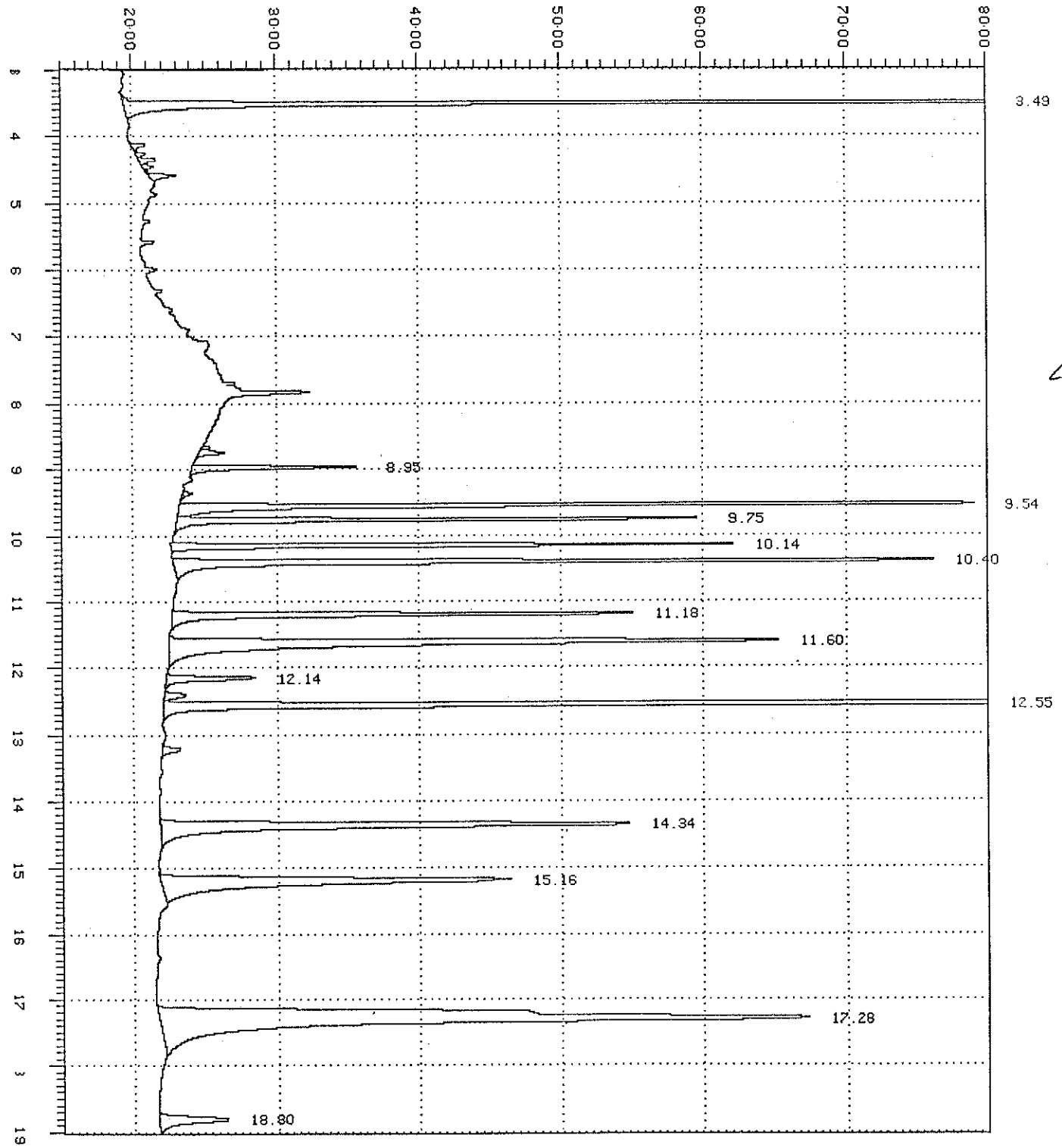
Method..... HERBUS1
Run sequence..... N0507
Calibration..... N0507
Internal standard calibration using area
Calibration last modified on 7-MAY-1992 at 22:18

Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[HRB05_1] 63 N07MAY92,5,1

HRB CSA 5% T=ST #5897. Amount : 1.000.
Inst.N/HP5890-Dual ECD Columns/RTx-5 .25um,30m,.25mm
Acquired on 7-MAY-1992 at 16:53
Reported on 7-MAY-1992 at 22:20
Box 1 (of 1)



L.L 5/7/92

Injection Report

Acquired on 7-MAY-1992 at 16:53

RMAL Chromatography System on DENCR3

Analyst Name : SMCCOOL
Lims Id : 2uL INJECTED
Comment : HP5890 Dual ECD Columns/RTx-5,RTx-20,0.25um,30m,0.25mm id
Method Title : METHOD FOR HERBICIDES
Sample Name : HRB CSA 5% T=ST #5897
Sample Id : VER # 5897
Sample Type : Standard Amount=1.00000
Bottle No : 5

L.C 5/7/92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
2	4.044	4.044	145208	342425	0.26370	Dalapon	2.1
7	11.138	11.138	87713	273091	0.13413	DCAA	2.7
8	11.551	11.551	59077	162170	0.02523	Dicamba	2.9
9	11.804	11.804	57203	136237	26.55811	MCPP	2.7
10	12.471	12.471	76838	233503	27.66571	MCPA	3.2
12	13.449	13.449	49867	185261	0.06584	Dichlorprop	3.5
13	13.907	13.907	140489	475668		D-BOB	3.5
14	14.502	14.502	71744	421693	0.13340	2,4-D	5.1
17	17.756	17.756	57254	336785	0.02608	Silvex	5.3
19	19.587	19.587	44103	316141	0.02517	2,4,5-T	6.1
20	21.702	21.702	61851	442473	0.06326	Dinoseb	6.4
21	22.147	22.147	44998	394129	0.25528	2,4-DB	7.5

Totals			
Unknowns	24870	119765	N/A
Quantified	896346	3719576	55.21591
Grand Total	921215	3839341	55.21591

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

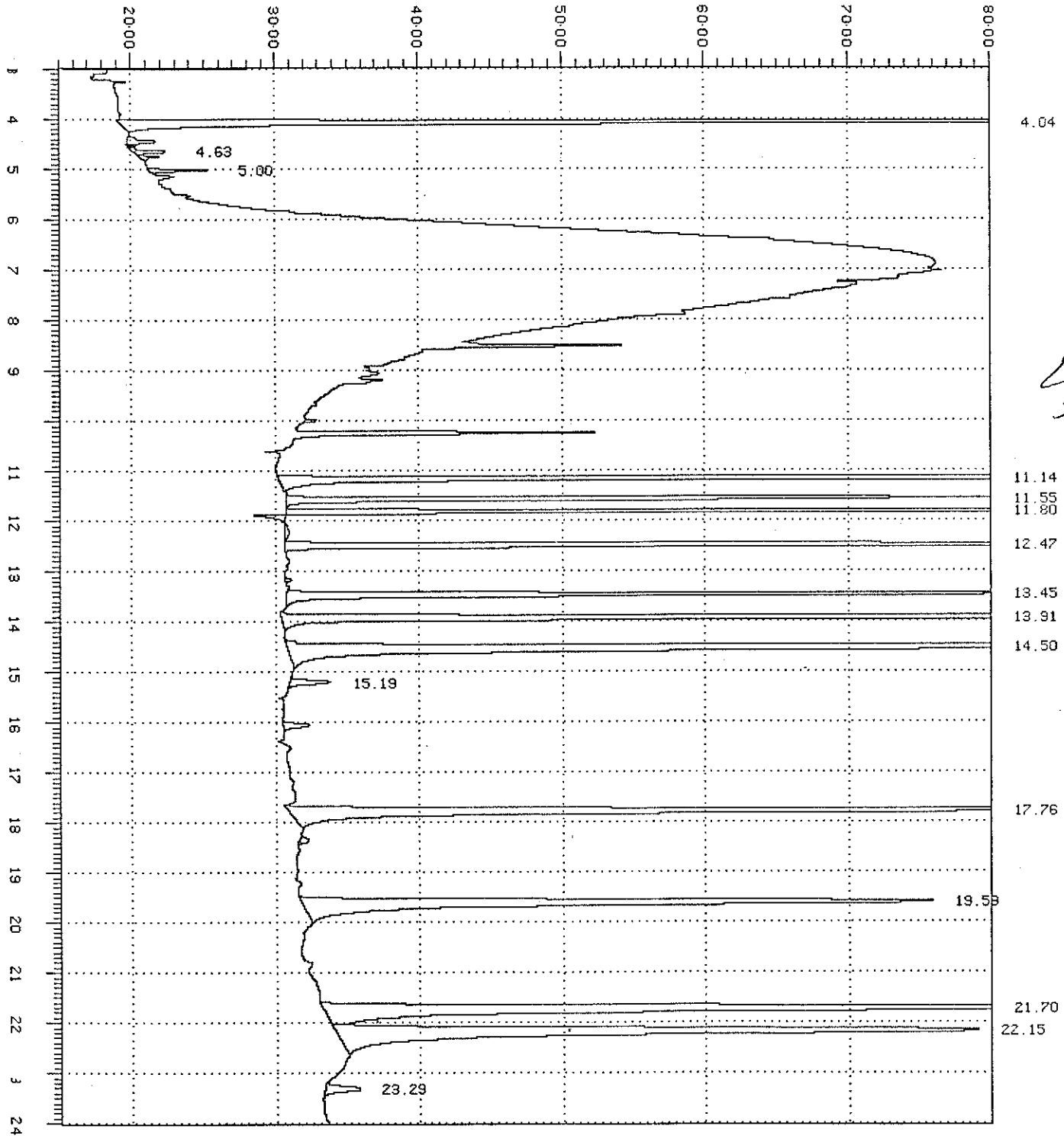
Method..... HERBUS1
Run sequence..... N0507
Calibration..... N0507
Internal standard calibration using area
Calibration last modified on 7-MAY-1992 at 22:17

Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[HRB05_1] 64 N07MAY92,5,1

HRB CSA 5% T=ST #5897. Amount : 1.000.
Inst.N/HP5890-Dual ECD Columns/RTx-5 .25um,30m,.25mm
Acquired on 7-MAY-1992 at 16:53
Reported on 7-MAY-1992 at 22:22
Box 1 (of 1)



LC
5/7/92

Injection Report

Acquired on 7-MAY-1992 at 17:31

RMAL Chromatography System on DENCR3

Analyst Name : SMCCOOL
Lims Id : 2uL INJECTED
Comment : HP5890 Dual ECD Columns/RTx-5,RTx-20,0.25um,30m,0.25mm id
Method Title : METHOD FOR HERBICIDES
Sample Name : HRB CSA 4% T=ST #5897
Sample Id : VER # 5897
Sample Type : Standard Amount=1.00000
Bottle No : 6

L.L 5/7/92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
2	3.507	3.493	77211	153799	0.21553	Dalapon	2.7A
11	9.547	9.538	45439	120186	0.11114	DCAA	2.7
12	9.764	9.751	28942	69797	0.02170	Dicamba	2.7
13	10.151	10.142	32452	76281	21.65636	MCPP	2.7
14	10.400	10.396	44391	133468	21.90292	MCPA	2.9
15	11.196	11.182	26345	83288	0.05537	Dichlorprop	2.9
16	11.618	11.604	33540	184988	0.11000	2,4-D	4.8
19	12.556	12.547	82674	238268		D-BOB	2.9
21	14.351	14.338	26171	133855	0.02090	Silvex	4.5
22	15.178	15.164	20326	135664	0.02102	2,4,5-t	5.6
C1		17.210	61782	390920	0.54291	2,4-DB/Dinoseb	
23	17.204	17.210	22786	98407	0.10073	- C1	5.3A
24	17.298	17.210	38996	292513	0.39415	- C1	6.4A

Totals			
Unknowns	29045	95583	N/A
Quantified	479272	1720513	44.65786
Grand Total	508318	1816095	44.65786

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

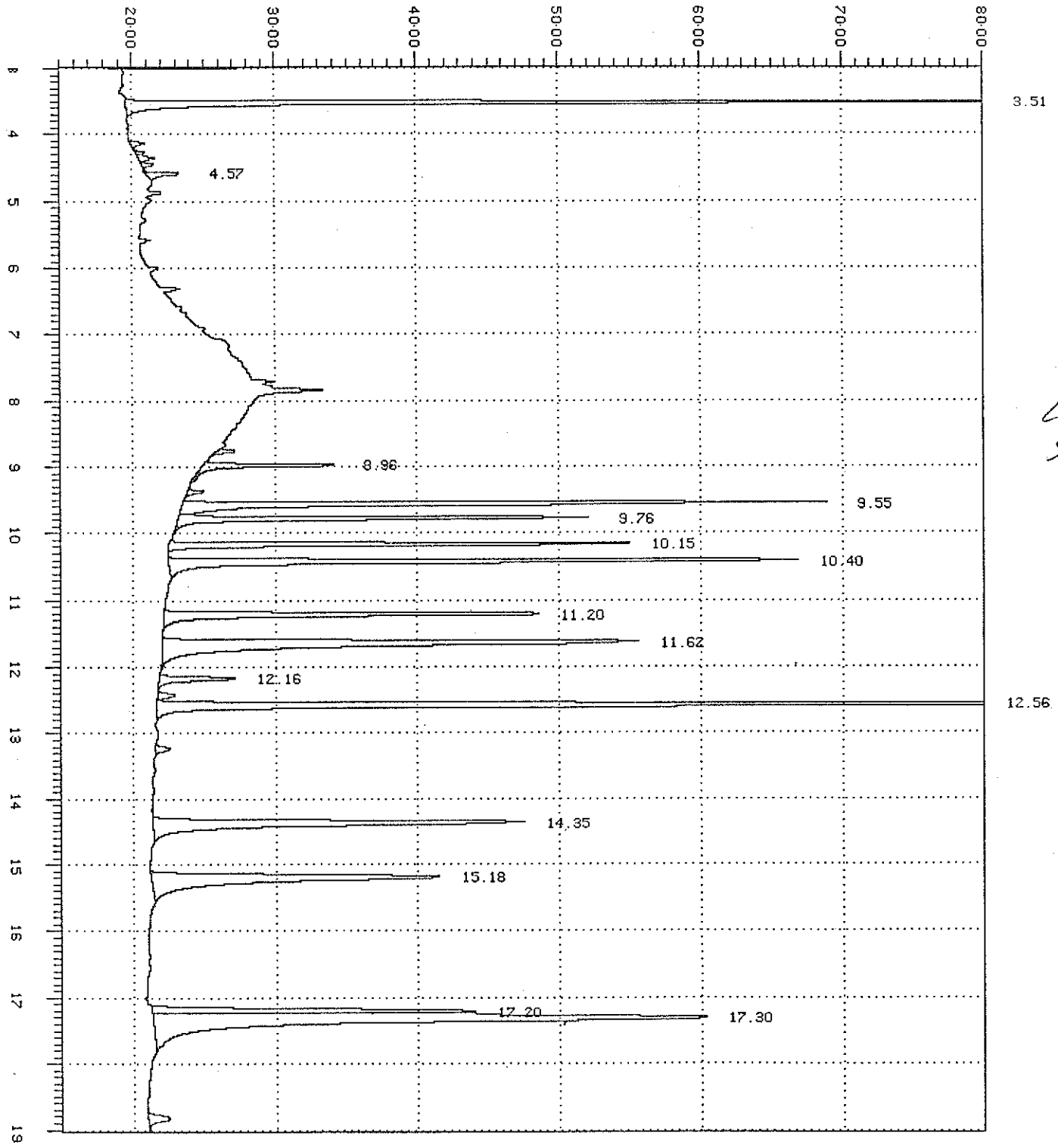
Method..... HERBUS1
Run sequence..... N0507
Calibration..... N0507
Internal standard calibration using area
Calibration last modified on 7-MAY-1992 at 22:18

Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[HRB05_1] 63 N07MAY92,6,1

HRB CSA 4% T=ST #5897. Amount : 1.000.
Inst.N/HP5890-Dual ECD Columns/RTx-5 .25um,30m,.25mm
Acquired on 7-MAY-1992 at 17:31
Reported on 7-MAY-1992 at 22:21
Box 1 (of 1)



L.L.
5/7/92

Injection Report

Acquired on 7-MAY-1992 at 17:31

RMAL Chromatography System on DENCR3

Analyst Name : SMCCOOL
Lims Id : 2uL INJECTED
Comment : HP5890 Dual ECD Columns/RTx-5,RTx-20,0.25um,30m,0.25mm id
Method Title : METHOD FOR HERBICIDES
Sample Name : HRB CSA 4% T=ST #5897
Sample Id : VER # 5897
Sample Type : Standard Amount=1.00000
Bottle No : 6

L.L 5/7/92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
2	4.036	4.044	127412	283696	0.21173	Dalapon	2.1
7	11.129	11.138	72627	235460	0.11314	DCAA	2.9
8	11.542	11.551	48640	153570	0.02397	Dicamba	2.9
9	11.791	11.804	45409	104290	21.65260	MCPP	2.4
10	12.462	12.471	62914	190424	23.43858	MCPA	3.2
11	13.440	13.449	41290	152198	0.05336	Dichlorprop	3.5
12	13.898	13.907	136099	472672		D-BOB	3.5
13	14.498	14.502	58034	349748	0.10795	2,4-D	5.6
16	17.747	17.756	47631	277305	0.02121	Silvex	5.3
17	19.578	19.587	36545	269501	0.02149	2,4,5-T	6.4
18	21.693	21.702	56072	390200	0.05536	Dinoseb	6.4
19	22.133	22.147	40668	345129	0.22177	2,4-DB	7.2

Totals			
Unknowns	26075	116143	N/A
Quantified	773340	3224193	45.92116
Grand Total	799415	3340336	45.92116

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

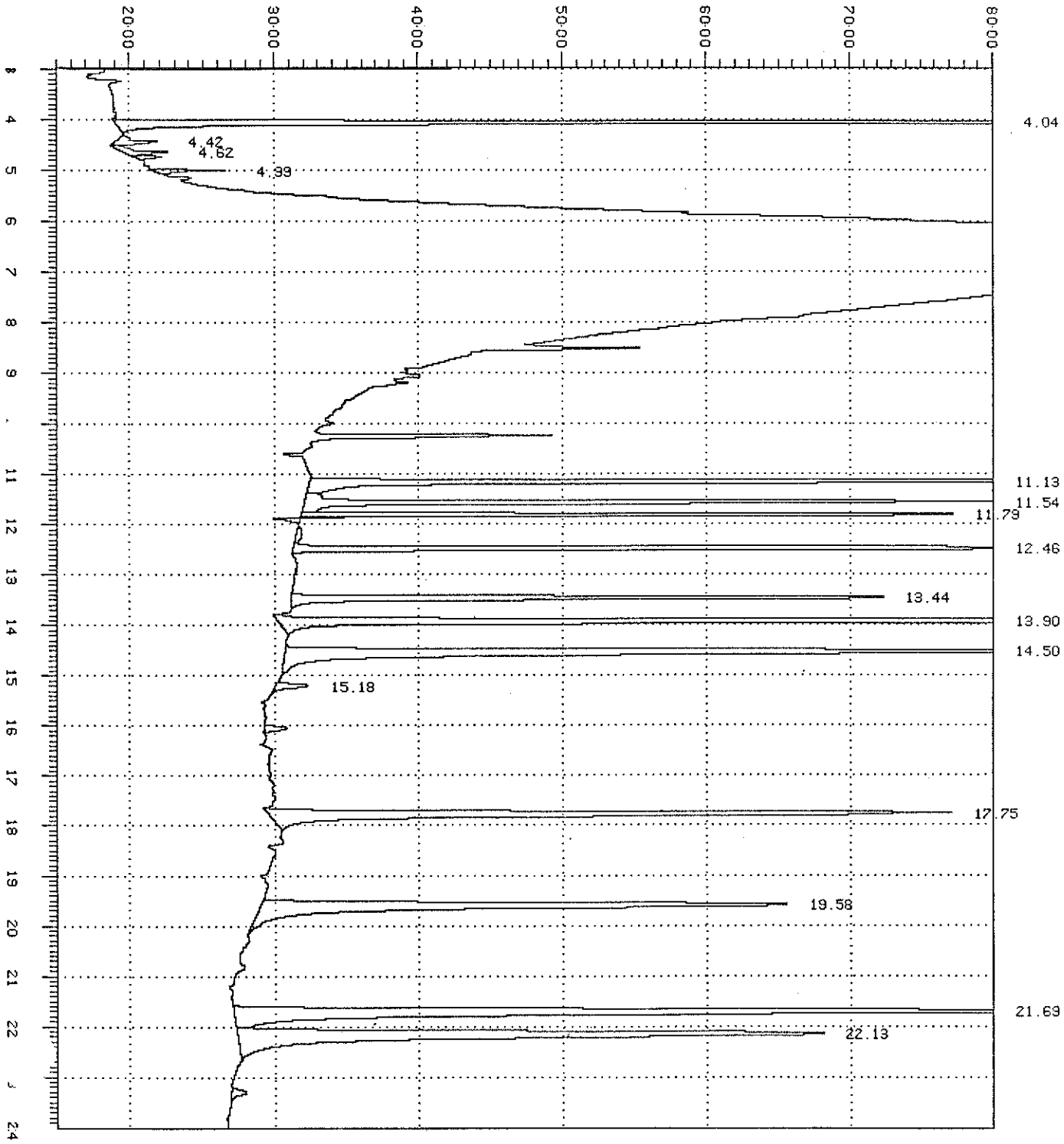
Method..... HERBUS1
Run sequence..... N0507
Calibration..... N0507
Internal standard calibration using area
Calibration last modified on 7-MAY-1992 at 22:17

Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[HRB05_1] 64 N07MAY92,6,1

HRL CSA 4% T=ST #5897. Amount : 1.000.
Inst.N/HP5890-Dual ECD Columns/RTx-5 .25um,30m,.25mm
Acquired on 7-MAY-1992 at 17:31
Reported on 7-MAY-1992 at 22:22
Box 1 (of 1)



L.C
5/7/92

Injection Report

Acquired on 7-MAY-1992 at 18:08

RMAL Chromatography System on DENCR3

Analyst Name : SMCCOOL
Lims Id : 2uL INJECTED
Comment : HP5890 Dual ECD Columns/RTx-5,RTx-20,0.25um,30m,0.25mm id
Method Title : METHOD FOR HERBICIDES
Sample Name : HRB CSA 2% T=ST #5897
Sample Id : VER # 5897
Sample Type : Standard Amount=1.00000
Bottle No : 7

L.L 5/7/92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
2	3.511	3.493	38855	85108	0.09515	Dalapon	2.1
11	9.551	9.538	23113	67724	0.04658	DCAA	2.9
12	9.769	9.751	14446	36771	0.00952	Dicamba	2.7
13	10.151	10.142	17170	41291	9.54900	MCPP	2.4
14	10.404	10.396	24438	83277	9.45478	MCPA	3.2
15	11.200	11.182	13350	44851	0.02346	Dichlorprop	3.2
16	11.631	11.604	16516	102294	0.04689	2,4-D	5.3
19	12.560	12.547	85543	251708		D-BOB	2.9
21	14.360	14.338	12195	67201	0.00979	Silvex	4.8
22	15.196	15.164	9541	66494	0.00982	2,4,5-t	5.9
C1		17.210	20400	200270	0.23855	2,4-DB/Dinoseb	
23	17.302	17.210	20400	200270	0.23855	- C1	9.9

Totals			
Unknowns	24172	98180	N/A
Quantified	275566	1046987	19.48354
Grand Total	299739	1145167	19.48354

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

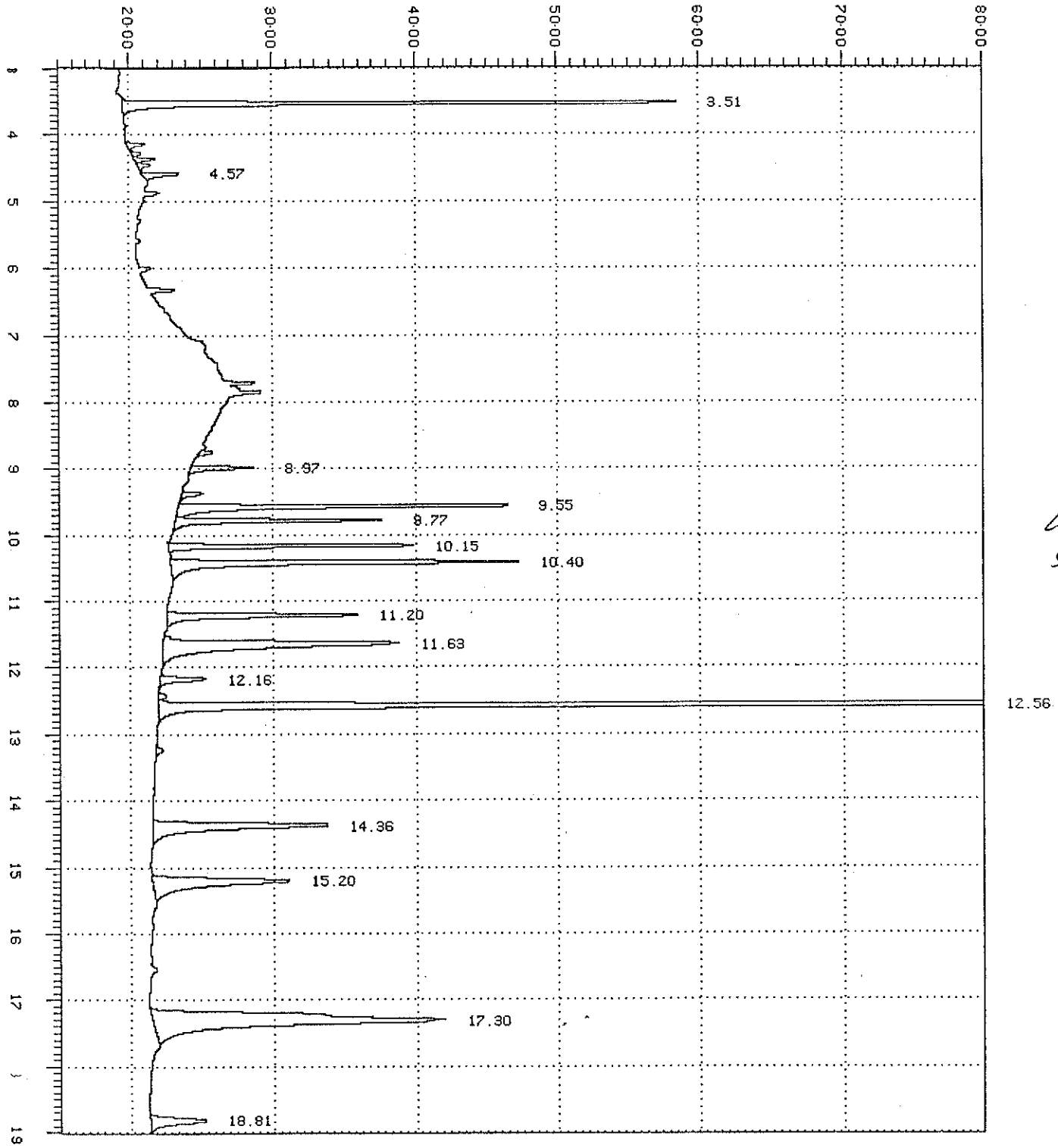
Method..... HERBUS1
Run sequence..... N0507
Calibration..... N0507
Internal standard calibration using area
Calibration last modified on 7-MAY-1992 at 22:18

Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[HRB05_1] 63 N07MAY92,7,1

HRL CSA 2% T=ST #5897. Amount : 1.000.
Inst.N/HP5890-Dual ECD Columns/RTx-5 .25um,30m,.25mm
Acquired on 7-MAY-1992 at 18:08
Reported on 7-MAY-1992 at 22:21
Box 1 (of 1)



L.L
5/7/92

Injection Report

Acquired on 7-MAY-1992 at 18:08

RMAL Chromatography System on DENCR3

Analyst Name : SMCCOOL
Lims Id : 2uL INJECTED
Comment : HP5890 Dual ECD Columns/RTx-5,RTx-20,0.25um,30m,0.25mm id
Method Title : METHOD FOR HERBICIDES
Sample Name : HRB CSA 2% T=ST #5897
Sample Id : VER # 5897
Sample Type : Standard Amount=1.00000
Bottle No : 7

L.L. 5/7/92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
2	4.044	4.044	66010	160135	0.09571	Dalapon	2.1
7	11.138	11.138	38727	122615	0.04594	DCAA	2.9
8	11.547	11.551	24391	65636	0.00911	Dicamba	2.9
9	11.791	11.804	18904	27588	9.47488	MCPP	2.4
10	12.462	12.471	31286	48587	8.94384	MCPA	3.2
12	13.444	13.449	21578	77593	0.02366	Dichlorprop	3.5
13	13.902	13.907	138808	480941		D-BOB	3.5
14	14.507	14.502	29526	187657	0.04722	2,4-D	5.9
17	17.756	17.756	23686	142999	0.00961	Silvex	5.6
18	19.591	19.587	18176	127947	0.00966	2,4,5-T	5.9
19	21.698	21.702	29129	195439	0.02372	Dinoseb	6.1
20	22.147	22.147	20802	171460	0.09457	2,4-DB	7.2

Totals			
Unknowns	24607	144868	N/A
Quantified	461024	1808600	18.77791
Grand Total	485631	1953468	18.77791

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

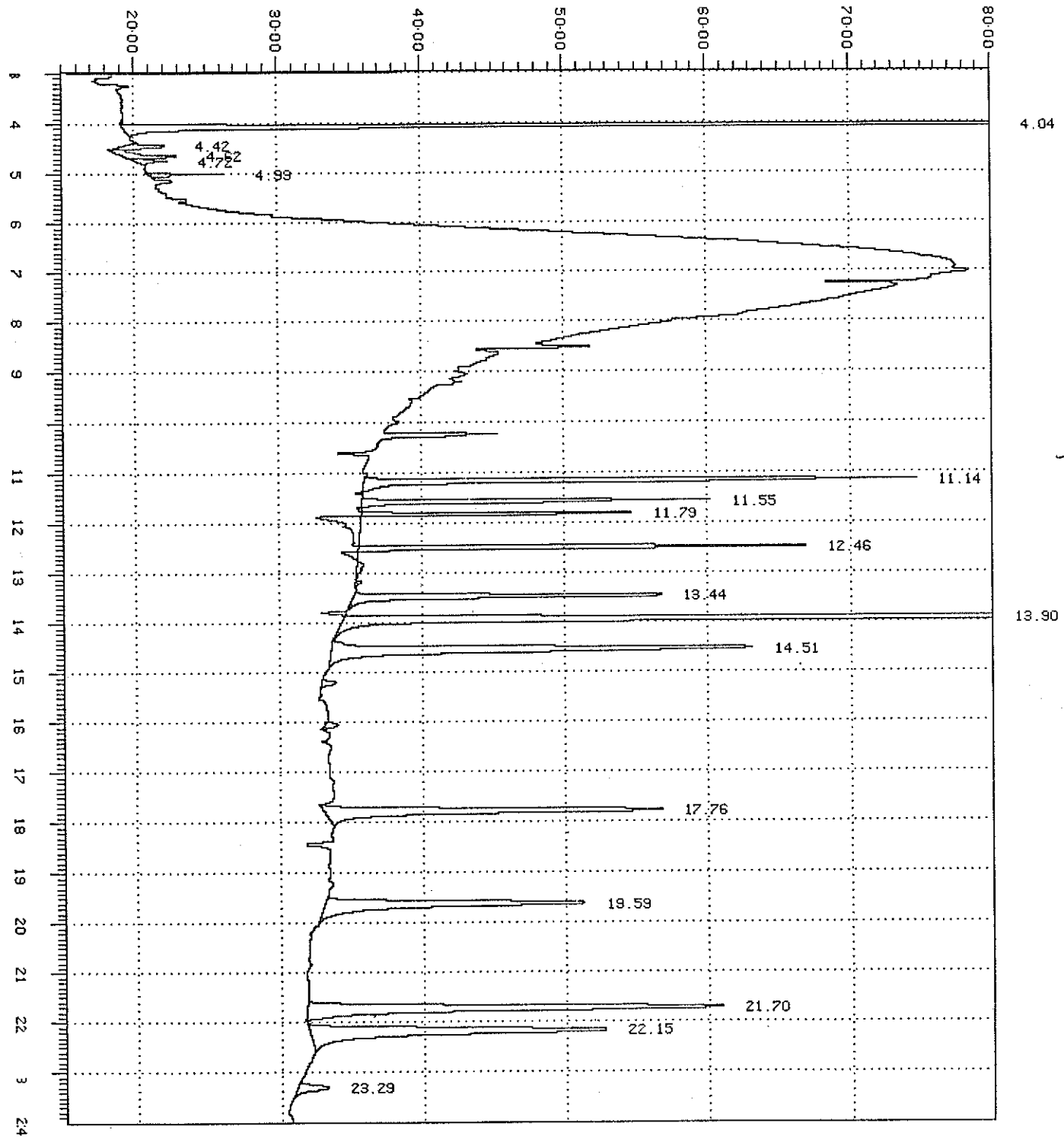
Method..... HERBUS1
Run sequence..... N0507
Calibration..... N0507
Internal standard calibration using area
Calibration last modified on 7-MAY-1992 at 22:17

Uncalibrated peaks use user factor (0.0000)

RMAL Chromatography System on DENCR3

[HRB05_1] 64 N07MAY92,7,1

HRb CSA 2% T=ST #5897. Amount : 1.000.
Inst.N/HP5890-Dual ECD Columns/RTx-5 .25um,30m,.25mm
Acquired on 7-MAY-1992 at 18:08
Reported on 7-MAY-1992 at 22:23
Box 1 (of 1)



L.C
5/7/92

RECEIVED
JAN 13 1993
QUALITY ASSURANCE SECTION
ENVIRONMENTAL SCIENCES DIV.

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC OPP SCS Stock		Page 1 of 2
SOP No.: LS-RMA-2500	Revision No.: 2.0	Effective Date 10/19/92
Supersedes: Original		

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1. TYPE OF STANDARD: Sample control spike stock

2. SOLVENT/MATRIX: Methanol

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Heather Esbenson	Date: 10/19/92
Management Approval: [signature]	Date: 10-22-92
QA Officer Approval: [signature]	Date: 10/22/92

3.2 To a 10.0mL volumetric flask add approximately 5 mL of methanol. Using a 1.0 mL Hamiltion gas tight syringe add 100 uL each of Chlormefos and Ethyl Pirmifos stocks at 10000 ug/ml in toluene to the flask. Bring to volume with methanol. Transfer to a 12mL amber vial.

4. SHELF LIFE: 3 months

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
Chlormefos (10000 ug/ml)	100.0 ug/ml
Ethyl Pirimifos (10000 ug/ml)	100.0 ug/ml

7. NOTES:

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC OPP SCS		Page _1_ of _2_
SOP No.: LS-RMA-2501	Revision No.: 2.0	Effective Date 10/19/92
Supersedes: Original		

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1. TYPE OF STANDARD: Sample control spike

2. SOLVENT/MATRIX: Methanol

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Heather Esbenson	Date: 10/19/92
Management Approval:	Date: 10-22-92
QA Officer Approval:	Date: 10/22/92

SOP No.:	Revision No.:	Effective Date:
LS-RMA-2501	2.0	10/19/92

3.2 To a 25.0mL volumetric flask add approximately 20 mLs of methanol. Using a 1.0 mL Hamiltion gas tight syringe add 500 uL of OPP SCS Stock at 100 ug/ml to the flask. Bring to volume with methanol. Transfer to a 1oz bottle.

4. SHELF LIFE: 3 months

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
OPP SCS Stock	
Chlormefos (100.0 ug/ml)	2.0 ug/ml
Ethyl Pirimifos (100.0 ug/ml)	2.0 ug/ml

7. NOTES:

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC OPP DCS Stock		Page _1_ of _2_
SOP No.: LS-RMA-2502	Revision No.: 2.0	Effective Date 10/19/92
Supersedes: Original		

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1. TYPE OF STANDARD: Control spike stock

2. SOLVENT/MATRIX: Methanol

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Heather Esbenson	Date: 10/19/92
Management Approval: [signature]	Date: 10-22-92
QA Officer Approval: [signature]	Date: 10/22/92

3.2 To a 10.0mL volumetric flask add approximately 5 mL of methanol. Using a 1.0 mL Hamiltion gas tight syringe add 100 uL each of the following stocks at 10000 ug/ml in toluene to the flask. Bring to volume with methanol. Transfer to a 12mL amber vial.

4. SHELF LIFE: 3 months

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
Ethion (10000 ug/ml)	100.0 ug/ml
Ethyl Parathion (10000 ug/ml)	100.0 ug/ml
Diazinon (10000 ug/ml)	100.0 ug/ml
Malathion (10000 ug/ml)	100.0 ug/ml
Methyl Parathion (10000 ug/ml)	100.0 ug/ml
Phorate (10000 ug/ml)	100.0 ug/ml

7. NOTES:

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC OPP DCS		Page 1 of 2
SOP No.: LS-RMA-2503	Revision No.: 2.0	Effective Date 10/19/92
Supersedes: Original		

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1. TYPE OF STANDARD: Control spike

2. SOLVENT/MATRIX: Methanol

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Heather Esbenson	Date: 10/19/92
Management Approval: Mark D. [illegible]	Date: 10-22-92
QA Officer Approval: Lee B. [illegible]	Date: 10/22/92

3.2 To a 25.0mL volumetric flask add approximately 20 mLs of methanol. Using a 1.0 mL Hamiltion gas tight syringe add 250 uL of OPP DCS Stock at 100 ug/ml to the flask. Bring to volume with methanol. Transfer to a 1oz bottle.

4. SHELF LIFE: 3 months

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
OPP DCS Stock	
Ethion (100 ug/ml)	1.0 ug/ml
Ethyl Parathion (100 ug/ml)	1.0 ug/ml
Diazinon (100 ug/ml)	1.0 ug/ml
Malathion (100 ug/ml)	1.0 ug/ml
Methyl Parathion (100 ug/ml)	1.0 ug/ml
Phorate (100 ug/ml)	1.0 ug/ml

7. NOTES:

STANDARD
OPERATING
PROCEDURE

Subject or Title: GC OPP AP9 CSA Stock		Page _1_ of _2_
SOP No.: LS-RMA-2504	Revision No.: 2.0	Effective Date 10/19/92
Supersedes: Original		

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1. TYPE OF STANDARD: Calibration standard stock

2. SOLVENT/MATRIX: Toluene

3. PREPARATION NOTES:

   3.1 Standards made for the extraction laboratory (DCSs, SCSs, MS/MSDs) and the instrumentation laboratory (calibration standards, check standards) must each be prepared from different sources. The other source may be another chemical supplier, the EPA, or the National Institute of Standards and Technologies (NIST). Refer to SOP: LP-RMA-0019.

Prepared by: Heather Esbenson	Date: 10/19/92
Management Approval: [signature]	Date: 10-22-92
QA Officer Approval: [signature]	Date: 10/22/92

3.2 To a 5.0mL volumetric flask add approximately 3 mLs of toluene. Using a 1.0 mL Hamiltion gas tight syringe add 500 uL each of the following stocks at 4000 ug/ml and the Ultra Scientific Opp Pesticide Mix at 2000 ug/ml to the flask. Bring to volume with toluene. Transfer to a 12mL amber vial.

4. SHELF LIFE: 3 months

5. SAFETY ISSUES: Refer to the MSDS's.

6. CONSTITUENTS/CONCENTRATION:

CHEMICAL/STOCK NAME (Conc., open date, etc.)	FINAL CONC.
Opp Pest. Mix (Ultra 2000 ug/ml)	
Methyl Parathion	200.0 ug/ml
Phorate	200.0 ug/ml
Dimethoate	200.0 ug/ml
Disulfoton	200.0 ug/ml
Famphur	200.0 ug/ml
Parathion	200.0 ug/ml
Thionazin	200.0 ug/ml
Sulfotepp	200.0 ug/ml
0,0,0-Triethyl Phosphorothioate	200.0 ug/ml
Ethyl Pirmifos (4000 ug/ml)	400.0 ug/ml
Chlormefos (4000.0 ug/ml)	400.0 ug/ml

7. NOTES:

SOP Number: LM-RMA-4006
Title: Organophosphorous Pesticides by Flame Photometric Gas Chromatography

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to EPA letters dated March 17, 1992 and September 15, 1992 to CWM containing comments (with additional attachments) regarding the CWM-Vickery GWMP QAPjP.

Section 6.18: Enseco-RMAL performs GC analyses using two dissimilar columns in order to accurately identify and quantitate organic compounds. Both columns are individually calibrated and must conform to constraints of ±20% difference for all compounds. When a compound is detected on both columns, the quantitative result is generally chosen from the lower of the two determinations. The higher value can reflect contamination from co-eluters, so the result is generally biased high. Based on this convention, Enseco-RMAL cannot designate one column as the primary column. Since quantitative data is taken from both columns throughout an analysis, both columns are, in a sense, equal to each other during the analysis. Only in extenuating circumstances will deviation from this procedure be this procedure be allowed. In those instances, the scientist will use his/her judgement to make accurate determinations.

Section 7.: Additional data requested by USEPA on Reagents and Standards is not routinely provided with analytical results. This information is available for review by regulatory agencies during external audits. A description of the materials provided in raw data packages (provided on client request) is found in QAPjP Appendix B-10A. QAPjP Appendix B-10C contains SOPs for the preparation of initial and continuing calibration standards,surrogates and matrix spikes.

Section 8.2.4 and 8.2.5: The requested information on surrogate and matrix spiking solutions is normally contained on the preparation sheets, which are located within a raw data package. QAPjP Appendix B-10C contains SOPs for the preparation of initial and continuing calibration standards, surrogates and matrix spikes.

Section 10: Linear dynamic range of compounds is determined from the instrument calibration, and normally covers one order of magnitude of analyte concentration. See QAPjP Appendix B-10B for an example of a five point instrument calibration. OPP target compound linear ranges are presented in a table at the front of QAPjP Appendix B-10B.

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to EPA letters dated March 17, 1992 and September 15, 1992 to CWM containing comments (with additional attachments) regarding the CWM-Vickery GWMP QAPjP.

Continuing calibration checks are performed every 10 investigative samples. Check standards are referenced in the SOP in Section 9.1, under calibration.

Section 10.1.2: Matrix spike and matrix spike duplicate analysis are performed at a frequency of one per every group of 20 investigative samples.

Section 11.4: See QAPjP Appendices C-3 and G for an example of the data reporting package, and QAPjP Appendix B-10A for raw data package content according to package type. Long form packages will be used.

Parameter retention time is variable with instrument condition and may vary slightly from calibration to calibration. Target parameters and relative retention times are provided in a table following this addendum page. Retention times recorded for an actual calibration are found in QAPjP Appendix B-10B.

Injection Report

Acquired on 10-OCT-1992 at 07:13

RMAL Chromatography System on DENCR3

Analyst Name : R.ECKERT
Lims Id : 2 UL INJECT
Comment : D-5890_A, DUAL FPD, RTX-1/RTX-5, 0.25uM, 30 M, 0.25 MM ID
Method Title : OPP 8140
Sample Name : 8140 CSA 0.2% #6355
Sample Id : CHK2 OPPA
Sample Type : Sample Amount=1.000000
Bottle No : 19

RE
10-13-92

PEAK INFORMATION

Peak	RT mins	RT Esc	Hght uV	Area uVs	ug/mL	Peak name	Width
1	5.707	0.000	635	1743	0.00000		4.3
2	5.947	0.000	866	950	0.00000		5.3
3	6.933	6.924	179106	418317	0.10124	DICHLORVOS	4.3A
4	7.213	0.000	5879	23036	0.00000		4.3
5	7.733	0.000	8377	20106	0.00000		2.1A
6	8.027	0.000	1558	8269	0.00000		4.3
7	8.302	8.293	458620	1350775	0.16709	PHOSDRIN	3.7
8	8.444	8.436	304152	300747	0.22113	CHLORMEFOS	2.1A
9	9.262	0.000	3197	14759	0.00000		5.2A
10	9.582	0.000	1020	2991	0.00000		4.3
11	9.876	0.000	2769	12832	0.00000		5.9
12	10.258	0.000	9039	25332	0.00000		3.2A
13	10.524	10.516	80564	257672	0.10228	ETHOPROP	3.7
14	10.800	10.791	134495	428697	0.54363	NALED	4.3
15	11.200	11.200	156143	462192	0.10274	SULFOTEPP	4.3A
16	11.404	11.396	76536	242047	0.10315	PHORATE	4.3
17	11.671	11.636	30228	165186	0.09397	DIMETHOATE	5.3
18	11.867	11.849	33094	182474	0.10748	DEMETON (O,S)	4.3
19	13.129	0.000	1095	3553	0.00000		4.3
C1		13.600	72331	458327	0.20593	DIAZINON/DISYSTON	
20	13.564	13.600	72331	458327	0.20593	- C1	6.4
21	14.124	0.000	986	1747	0.00000		2.1A
22	15.173	0.000	1268	6364	0.00000		6.9
23	15.431	15.413	35847	213349	0.09907	METHYL PARATHION	5.9
24	16.196	0.000	866	2906	0.00000		5.9
25	16.711	16.702	36039	207467	0.09914	RONNEL	5.9
26	18.142	18.133	79646	419694	0.24729	MALATHION	5.9
27	18.498	18.480	35511	218236	0.09969	FENTHION	5.9
28	18.764	18.747	32900	194833	0.09851	ETHYL PARATHION	6.9
29	18.898	18.889	32705	200301	0.09517	CHLORPYRIFOS	6.9
30	20.053	20.053	31638	209551	0.11081	TRICHLORONATE	6.9
31	20.907	20.898	71008	432378	0.20993	ETHYL PIRIMIPHOS	6.4
32	22.053	22.044	29521	176500	0.24105	MERPHOS	5.9

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
33	23.431	0.000	2232	10638	0.00000		5.3
34	23.724	23.707	48764	326585	0.24305	TETRACHLORVINPHOS	6.9
35	25.484	25.476	24306	158193	0.09423	PROTHIOPHOS	6.9
36	26.169	26.160	69151	449893	0.27353	DEF	6.9
37	27.547	0.000	1259	14832	0.00000		10.7
38	28.116	28.107	94137	590229	0.44570	FENSULFOTHION	5.9
39	29.138	29.138	78941	295545	0.09800	ETHION	4.3
40	29.529	29.529	39353	153419	0.09527	SULPROFOS	4.3
41	30.267	0.000	753	2463	0.00000		6.9
42	30.462	0.000	877	2825	0.00000		4.8
43	30.791	30.791	53549	160794		TRIPHENYLPHOSPHATE	4.3A
44	32.187	32.178	112360	380465	0.46044	METHYL AZINPHOS	3.7
45	33.538	0.000	1342	5204	0.00000		4.8
46	33.929	33.920	27660	113024	0.22142	COUMAPHOS	4.3

Totals			
Unknowns	0	0	N/A
Quantified	2501356	9847938	4.98707
Grand Total	2501356	9847938	4.98707

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... OPP
Run sequence..... D1009
Calibration..... OPP1009
Internal standard calibration using area
Calibration last modified on 13-OCT-1992 at 10:44

Uncalibrated peaks use user factor (0.0000)

B-10

Injection Report

Acquired on 10-OCT-1992 at 07:13

RMAL Chromatography System on DENCR3

Analyst Name : R.ECKERT
Lims Id : 2 UL INJECT
Comment : D-5890_A, DUAL FPD, RTX-1/RTX-5, 0.25uM, 30 M, 0.25 MM ID
Method Title : OPP 8140
Sample Name : 8140 CSA 0.2% #6355
Sample Id : CHK2 OPPA
Sample Type : Sample Amount=1.000000
Bottle No : 19

RE
10-13-92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
1	6.711	0.000	1155	4325	0.00000		4.3A
2	8.480	8.471	168526	443749	0.10209	DICHLORVOS	3.2A
3	8.844	0.000	5298	21662	0.00000		3.7
4	9.582	0.000	6050	20889	0.00000		4.3A
5	10.000	0.000	3022	14793	0.00000		5.3
6	10.480	10.471	484978	1553773	0.17029	PHOSDRIN	4.3A
7	10.747	10.738	190981	631423	0.21157	CHLORMEFOS	3.7
8	12.178	0.000	2398	12139	0.00000		5.3
9	13.351	0.000	2209	18941	0.00000		6.9
10	13.964	0.000	5046	23927	0.00000		4.3
11	14.551	14.533	55163	272502	0.10321	ETHOPROP	5.3
12	15.342	15.324	74150	402930	0.53702	NALED	5.3
13	15.991	15.973	95178	478794	0.10230	SULFOTEPP	5.3
14	16.373	16.364	43163	236004	0.10128	PHORATE	5.9
15	17.387	17.351	26950	156344	0.09788	DEMETON (O,S)	5.9
16	17.618	17.582	27528	223030	0.09595	DIMETHOATE	6.9
17	20.018	20.009	43671	251736	0.10149	DIAZINON	6.4
18	20.364	20.347	41530	267009	0.10555	DISYSTON	6.4
19	23.733	23.698	30952	239914	0.09447	METHYL PARATHION	7.5
20	24.880	24.858	31704	237517	0.10134	RONNEL	6.9
21	27.067	27.049	66348	494380	0.24608	MALATHION	7.5
22	27.742	27.724	38983	232370	0.09514	FENTHION	5.3A
C1		27.950	48176	480321	0.20212	CHLORPYRIFOS/ETHYL PARATHION	
23	27.929	27.950	48176	480321	0.20212	- C1	10.1
24	28.409	0.000	1268	6136	0.00000		5.9
25	28.640	28.631	44629	217367	0.09949	TRICHLORONATE	5.9
26	29.244	29.236	124869	464565	0.19558	ETHYL PIRIMIPHOS	4.3
27	29.724	0.000	62661	220663	0.00000		4.3A
28	30.320	0.000	1404	9993	0.00000		6.9
29	30.640	30.631	3581	18513	0.34542	MERPHOS	4.3A
30	31.004	30.996	105288	376046	0.24400	TETRACHLORVINPHOS	4.3
31	31.573	31.564	51094	167222	0.09332	PROTHIOPHOS	4.3
32	31.760	31.751	186330	591743	0.23418	DEF	3.7

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
33	32.809	32.800	145714	574101	0.42934	FENSULFOTHION	4.3
34	33.067	33.058	73844	276765	0.08823	ETHION	4.3
35	33.369	33.360	34215	140544	0.08941	SULPROFOS	4.3
36	34.382	34.373	46210	202070		TRIPHENYLPHOSPHATE	5.3
37	36.089	36.080	47487	256860	0.44143	METHYL AZINPHOS	5.9
38	37.209	0.000	765	5223	0.00000		6.9
39	37.893	37.884	12989	95787	0.24221	COUMAPHOS	7.5
40	39.147	0.000	657	1643	0.00000		1.6

Totals			
Unknowns	0	0	N/A
Quantified	2436053	10343720	4.36937
Grand Total	2436053	10343720	4.36937

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... OPP
Run sequence..... D1009
Calibration..... OPP10009
Internal standard calibration using area
Calibration last modified on 13-OCT-1992 at 10:51

Uncalibrated peaks use user factor (0.00000)

OPP Target Compound Linear Ranges

Compound	Instrument Levels Low Point	Instrument Levels High Point
8140 LIST:		
Dichlorvos (Vapona)	0.025ug/mL	0.250ug/mL
Phosdrin (Mevinphos)	0.0375ug/mL	0.375ug/mL
Chlormephos	0.050ug/mL	1.000ug/mL
Ethoprop	0.025ug/mL	0.250ug/mL
Naled (Dibrom)	0.125ug/mL	1.250ug/mL
Sulfotepp	0.025ug/mL	0.250ug/mL
Phorate (Thimet)	0.025ug/mL	0.250ug/mL
Demeton (O,S)	0.025ug/mL	0.250ug/mL
Dimethoate	0.025ug/mL	0.250ug/mL
Diazinon	0.025ug/mL	0.250ug/mL
Disyston	0.025ug/mL	0.250ug/mL
Methyl Parathion	0.025ug/mL	0.250ug/mL
Ronnel	0.025ug/mL	0.250ug/mL
Malathion	0.0625ug/mL	0.625ug/mL
Fenthion (Baytex)	0.025ug/mL	0.250ug/mL
Chlorpyrifos (Dursban)	0.025ug/mL	0.250ug/mL
Ethyl Parathion	0.025ug/mL	0.250ug/mL
Trichlornate	0.025ug/mL	0.250ug/mL
Ethyl Pirimphos	0.050ug/mL	1.000ug/mL
Merphos	0.0625ug/mL	0.625ug/mL
Tetrachlorvinphos(Stirophos)	0.0625ug/mL	0.625ug/mL
Prothiophos (Tokuthion)	0.025ug/mL	0.250ug/mL
DEF	0.0625ug/mL	0.625ug/mL
Fensulfothion	0.125ug/mL	1.250ug/mL
Ethion	0.025ug/mL	0.250ug/mL
Sulprofos (Bolstar)	0.025ug/mL	0.250ug/mL
Methyl Azinphos (Guthion)	0.125ug/mL	1.250ug/mL
Coumaphos	0.0625ug/mL	0.625ug/mL
APPENDIX IX LIST:		
o,o,o-TEPT	0.025ug/mL	0.250ug/mL
Thionazin	0.025ug/mL	0.250ug/mL
Sulfotepp	0.025ug/mL	0.250ug/mL
Phorate (Thimet)	0.025ug/mL	0.250ug/mL
Dimethoate	0.025ug/mL	0.250ug/mL
Disyston	0.025ug/mL	0.250ug/mL
Methyl Parathion	0.025ug/mL	0.250ug/mL
Ethyl Parathion	0.025ug/mL	0.250ug/mL
Famphur	0.025ug/mL	0.250ug/mL

ADDENDUM TO CWM Vickery GWMP QAPjP Appendix B-10
SOP Number: LM-RMA-4006
Title: Organophosphorous Pesticides by Flame Photometric Gas Chromatography

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This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to an EPA letter dated March 17, 1992 to CWM containing comments (with additional attachment) regarding the CWM-Vickery GWMP QAPjP.

Section 6.18: For the identification of a compound, Enseco requires mutual agreement that there is a detectable value on each column. The lower value from the two columns (above the reporting limit) is reported.

Section 7.: Additional data requested by USEPA on Reagents and Standards is not routinely provided with analytical results. This information is available for review by regulatory agencies during external audits. A description of the materials provided in raw data packages (provided on client request) is found in QAPjP Appendix B-10A.

Section 8.2.4 and 8.2.5: The requested information on surrogate and matrix spiking solutions is contained on the preparation sheets, which are located within a raw data package.

Section 10: Linear dynamic range of compounds is determined from the instrument calibration, and normally covers one order of magnitude of analyte concentration. See QAPjP Appendix B-10B for an example of a five point instrument calibration. The standard concentrations used for various analytes are listed as the "true" value, in ug/mL.

Continuing calibration checks are performed every 10 investigative samples. Check standards are referenced in the SOP in Section 9.1, under calibration.

Section 10.1.2: Matrix spike and matrix spike duplicate analysis are performed at a frequency of one per every group of 20 investigative samples.

Section 11.4: See QAPjP Appendix C-3 for an example of the data reporting package.

Parameter retention time is variable with instrument condition and may vary slightly from calibration to calibration. Retention times recorded for an actual calibration are found in QAPjP Appendix B-10B.

STANDARD
OPERATING
PROCEDURE

Subject or Title:
Organophosphorous Pesticides (EPA Method 614/8140)

SOP No.: LM-RMA-4006
Revision No.: 2.0
Effective Date: March 3, 1989

Supersedes: 1.0

Soil and Water
by Flame Photometric Gas Chromatography

1. Scope & Application

1.1 Analytes

Naled
Dichlorvos
Trichlorofon
Phosdrin (Mevinphos)
Thionazin
Demeton-S
Ethoprop
Sulfotep
Phorate (Thimet)
Dimethoate (Cygon)
Demeton-O
Propetamphos
Terbufos
Disulfoton (Disyston)
Diazinon
Methyl parathion
Ronnel
Fenthion
Guthion

Malathion
Parathion
Chlorpyrifos
Crotoxyphos
Tetrachlorvinphos
Tokuthion
Butifos (DEF)
Methyl trithion
Fensulfothion
Ethion
Famphur
Trithion
Phosmet
Azinphos methyl
Coumaphos
Dioxathion
0,0,0-
Triethylphosphorothioate
Carbophenothion
Dioxathion

Prepared by: Debra Henderer
Date: January 25, 1989

Management Approval: Susan M. Brillante
Date: 3/14/89

QA Officer Approval: Ian Eidenbarger
Date: 3/15/89

1.2 Detection Limits

1.2.1 The detection limits are readily achieved for most compounds. there are few exceptions, however. Dioxathion's detection limit is approximately 10 times higher and Famphur's is approximately 2 times higher.

1.2.2 Instrumental detection limits are o.05 ug/mL for most compounds.

1.3 Applicable matrices

1.3.1 Aqueous and solid samples may be analyzed by this method.

1.4 Linear Range of Instrument

1.4.1 Calibration standards are run at 5 levels and a calibration curve generated for each compound.

1.5 Analysis time

1.5.1 Sample prep requires one full day for completion.

1.5.2 Analysis times are typically approximately 40 minutes per sample or standard.

2. Summary of Method

2.1 Samples are extracted by shakeout or sonication using hexane and exchanged after solvent concentration to toluene. Analysis is performed by gas chromatography using a flame photometric detector which is operated in the phosphorous mode.

3. Comments

3.1 Interferences

3.1.1 Waters do not require clean up as there is not usually a problem with interferences. Because the detector is so selective, soil samples may be run without dilution even if they are colored. The greatest risk is contamination of the column or injector as the detector is somewhat robust.

4. Safety Issues

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound

must be treated as potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis.

5. Sample Collection, Preservation and Handling

5.1 Grab samples must be collected in glass containers. Conventional sampling practices should be followed; however, the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of plastic and other potential sources of contamination.

5.2 The samples must be iced or refrigerated at 4°C from the time of collection until extraction.

5.3 All samples must be extracted within 7 dy of collection and completely analyzed within 40 dy of extraction.

6. Apparatus and Materials

Sampling equipment, for discrete or composite sampling.

6.1 Grab sample bottle - Amber borosilicate or flint glass, 1 L or 1 qt volume, fitted with screw caps lined with TFE-fluorocarbon. Aluminum foil may be substituted for TFE if the sample is not corrosive. If amber bottles are not available, protect samples from light. The container and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

6.2 Automatic sampler (optional) - Must incorporate glass sample containers for the collection of a minimum of 250 mL. Samples containers must be kept refrigerated at 4°C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing must be thoroughly rinsed with methanol, followed by repeated rinsings with reagent water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

Glassware (All specifications are suggested. Catalog numbers are included for illustration only.)

6.3 Separatory funnel - 125 mL, 1000 mL and 2000 mL, with TFE-fluorocarbon stopcock, ground glass or TFE stopper.

6.4 Filtering funnel - 75 mm top diameter.

6.5 Chromatographic column - 400 mm long x 19 mm ID with coarse fritted disc at bottom and TFE-fluorocarbon stopcock (Kontes K-420540-0224 or equivalent).

6.6 Concentrator tube, Kuderna-Danish - 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

6.7 Evaporative flask, Kuderna-Danish - 500-mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

6.8 Snyder column, Kuderna-Danish - three-ball macro (Kontes K-503000-0121 or equivalent).

6.9 Pipet, disposable - 140 mm long x 5 mm ID.

6.10 Vials - 10 to 15 mL capacity with TFE-fluorocarbon lined screw cap.

6.11 Boiling chips - approximately 10/40 mesh. Heat at 400°C for 30 min or Soxhlet extract with CH_2Cl_2.

6.12 Water bath - Heated, with concentric ring cover, capable of temperature control (± 2°C). The bath should be used in a hood.

6.13 Balance - Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.14 Sonicator - A horn-type sonicator equipped with a titanium tip to be used. The following sonicator, or an equivalent brand and model, is recommended:

Ultrasonic cell distrupter: Heat Systems - Ultrasonics, Inc., Model W-385 (475 watt) sonicator or equivalent (Power wattage must be a minimu of 375 with pulsing capability and No. 200 1/2" Tapped Distrupter Horn) plus No. 207 3/4" Tapped Disrupter Horn, and No. 419 1/8" Standard Tapered microtip probe.

6.15 Sonabox - Recommended with above disrupters for decreasing cavitation sound (Heat Systems - Ultrasonics, Inc., Model 432B or equivalent).

6.16 Gas chromatograph - Analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector and stripchart recorder. A data system is recommended for measuring peak areas.

6.17 Detector - flame photometric detector operated in the Phosphorous mode (526 nm filter).

6.18 Column

Column 1	DB-5, 30 m long x 0.53 mm I.D., 1.5 u film thickness or equivalent.
Column 2	DB-608 30 m long x 0.53 mm I.D., 1.0 u film thickness or equivalent.

7. Reagent and Standards

7.1 Reagent water - Reagent water is defined as a water in which an interferent is not observed at the method detection limit of each parameter of interest.

7.2 Acetone, hexane, isooctane, methylene chloride, toluene, pesticide quality or equivalent.

7.3 Diethyl ether - nanograde, redistilled in glass if necessary. Must be free of peroxides as indicated by EM Quant test strips. (Available from Scientific Products Co., Cat. No. P1126-8, and other suppliers.) Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL ethyl alcohol preservative must be added to each liter of ether.

7.4 Acetonitrile, hexane-saturated - Mix pesticide quality acetonitrile with an excess of hexane until equilibrium is established.

7.5 Sodium sulfate - (ACS) granular, anhydrous. Dry in a shallow tray at 400°C for a minimum of 4 hr to remove phthalates and other interfering organic substances. Alternatively, heat 16 hr at 450-500°C in a shallow tray or Soxhlet extract with methylene chloride for 48 hr.

7.6 Sodium chloride solution, saturated - Prepare saturated solution of NaCl in reagent water and extract with hexane to remove impurities.

7.7 Alumina - Woelm, neutral; deactivate by pipeting 1 mL of distilled water into a 125 mL ground glass-stoppered Erlenmeyer flask. Rotate flask to distribute water over surface of glass. Immediately add 19.0 g fresh alumina through small powder funnel. Shake flask containing mixture for 2 hr on a mechanical shaker.

7.8 Florisil - PR grade (60/100 mesh). Purchase activated at 1250°F and store in dark in glass container with ground glass stopper or foil-lined screw cap. Before use activate each batch at least 16 h at 130°C in a foil covered glass container.

7.9 Primary standard solutions - Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions.

7.9.1 Prepare primary standard solutions by accurately weighing approximately 0.0100 g of pure material. Dissolve the material in pesticide quality isooctane or acetone and dilute to volume in a 10 mL volumetric flask. Larger volumes may be used at the convenience of the analyst. If compound purity is certified at 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

7.9.2 Transfer the stock standard solutions into TFE-fluorocarbon-sealed screw cap vials. Store at 4°C and protect from light. Frequently check primary standard solutions for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

7.9.3 Primary standard solutions must be replaced after 6 mo or sooner if comparison with check standards indicates a problem.

8. Procedure

8.1 Method Summary

This method is applicable to the determination of organophosphorus pesticides in aqueous samples. A 1 L water sample is extracted with CH_2Cl_2. The extract is dried, exchanged to toluene, and concentrated to a volume of 10.0 mL. Analysis is performed on a gas chromatograph equipped with a nitrogen phosphorous detector.

8.2 Extraction of Liquid Samples

8.2.1 If the sample contains particulate matter, transfer 3-4 mL of sample to a 12 mL screw top glass vial with a Teflon liner. Add approximately 1 mL of CH_2Cl_2. Shake vigorously for about 30 and allow the layers to separate. If no emulsion forms proceed with the shakeout technique (Part I). If an emulsion forms, use the continuous extractor method (Part II). In general, surrogate recoveries are erratic for samples extracted by continuous extractor, so it is only to be used in cases where there is an extreme amount of sediment or an extreme emulsion forms during the emulsion test.

NOTE: All glassware must be solvent rinsed with CH_2Cl_2 before use.

8.2.2 Samples that are clearly described as ground waters in the project folder are decanted from any sediment in the bottle. All others are mixed by shaking the bottle.

8.2.3 If the sample is in a 1 L or smaller bottle, mark the water meniscus on the side of the sample bottle for later determination of the sample volume. In this case, the volume will be determined after step 6. If the sample is in a larger bottle, use a 1 L graduated cylinder. Pour the sample into a 2 L separatory funnel. For blanks and quality control work, pour 1 L of carbon filtered water into the separatory funnel.

8.2.4 Add the appropriate surrogate standard to each sample, including blanks, using a disposable pipet. Make sure the pipet tip is below the surface of the sample while the standard is being added. Mix the sample immediately by stirring with the pipet. Note the addition of the standard, including who added it, the date and who made the standard on the prep sheet.

8.2.5 Add the spike mix, if appropriate, using the procedure described above.

8.2.6 Check the pH and note the initial pH on the prep sheet. Adjust the pH to 5-8, if necessary, with 10 N NaOH or 1:1 H_2SO_4. Note the adjusted pH on the prep sheet.

8.2.7 For samples that were mixed before extracting, add 60 mL CH_2Cl_2 to the sample bottle to rinse the inner walls. Do NOT cap and shake the bottle, rinse the glass only; transfer the solvent to the separatory funnel. For samples that were decanted, add the first aliquot of CH_2Cl_2 directly to the

separatory funnel. Extract the sample by shaking it for two minutes with frequent ventilation.

8.2.8 Allow the layers to separate. If there is an emulsion, break it.

8.2.9 Drain the bottom layer (CH_2Cl_2) into a 250 mL beaker.

8.2.10 Repeat the extraction twice more using a 60 mL aliquot of CH_2Cl_2 each time. Collect the solvent in the same beaker described in step 8. Record the volume recovered of the prep sheet.

8.2.11 Put a plug of glass wool in a funnel and fill about 2/3 full with Na_2SO_4. Rinse the funnel and Na_2SO_4 with 30-40 mL of CH_2Cl_2, discard. Pour the extract through the Na_2SO_4 into a 500 mL Kuderna-Danish (K-D) evaporative concentrator. Rinse the beaker then the Na_2SO_4 with small amounts of CH_2Cl_2. Add these rinses to the K-D.

8.2.12 Add a boiling chip to the K-D and attach a 3 ball Snyder column to the top. Pre-wet the column by adding about 1 mL of CH_2Cl_2 to the top.

NOTE: The concentration step is critical; losses of target compounds can occur if care is not taken.

8.2.13 Place the K-D in a hot water bath set at 95°C so that the receiver tube is immersed in hot water and the entire lower rounded surface is bathed in steam. At a proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 3-5 mL, remove the K-D from the bath and allow it to cool completely.

8.2.14 If the extract is highly colored or a precipitate forms during concentration, consult a supervisor. See for florisil column cleanup.

8.2.15 After the K-D has cooled, rinse the Snyder column and middle flask with a small amount of CH_2Cl_2. Transfer the extract to a calibrated 15 mL centrifuge tube, rinsing with a small amount of CH_2Cl_2. Be sure to rinse all of the ground glass joints well, as compounds collect on the ground glass.

8.2.16 Carefully concentrate the extract to approximately 2 mL under a gentle stream of nitrogen using the N-evap apparatus.

8.2.17 Add 2 mL of toluene and continue to evaporate the extract to slightly below the 2 mL mark on the tube. Remove the tube from the bath and allow it to completely cool.

8.2.18 Adjust the volume with toluene to 10.0 mL. If the extract is highly colored, forms a precipitate, or stops evaporating, consult a supervisor.

8.2.19 Transfer the extract to a labeled 12 mL screw-top vial with a Teflon lined cap, mark the meniscus, and place on a rack with other samples with the same project, number, if any.

8.2.20 Complete the prep sheet for the extraction and concentration steps.

8.3 Continuous Extractor Technique

8.3.1 See 8.2.1 for guidance on when to use the continuous extractor.

8.3.2 Put 250 mL CH_2Cl_2 in a round bottom flask, add a few boiling chips. Add 300 mL of CH_2Cl_2 to the extractor flask.

8.3.3 Carefully pour 500 mL of carbon filtered water into the extractor. Minimize the disturbance of the solvent layer and avoid getting water into either sidearm by pouring the water down the back of the extractor.

8.3.4 Open the sample container and remove a small aliquot of the sample with a pasteur pipet. Place enough of the sample on a pH paper to thoroughly soak the paper. Compare the pH paper with the chart on the container and record the initial pH on the prep sheet.

8.3.5 a: For water samples:

Decant ground water samples into the extractor, mix all other water samples by shaking the sample bottle before pouring. For samples in 1 L or smaller bottles, mark the meniscus on the side of the sample bottle and pour approximately 1 L of the sample into the extractor flask. Measure the exact volume by adding tap water to the bottle to the marked level and measuring the volume with a graduated cylinder. For samples in bottles larger than 1 L, measure 1 L of the sample in a graduated cylinder. Record the volume on the prep sheet.

b: For sludge samples:

Remove an aliquot of approximately 10 g, place it in a pre-weighed aluminum weigh boat and air-dry it overnight to determine the percent moisture. Weigh the bottle containing the sample and carefully transfer the sample to the extractor flask. Re-weigh the bottle, calculate the difference and record it as the amount extracted on the prep sheet.

8.3.6 Add enough carbon filtered water to the extractor to bring the level of the solvent in the removable sidearm to about 4 cm from its highest point. Make sure that the extractor stays in a strictly vertical position.

8.3.7 Add the appropriate surrogate standard to each sample using a disposable pipet. Make sure that the tip of the pipet is below the surface of the liquid in the extractor as the standard is being added. Mix the sample immediately using a glass stirring rod. Note the volume of the spike added and all necessary information from the label of the standard on the prep sheet.

8.3.8 Add the spike mix, if appropriate, using the procedure described in step 7.

8.3.9 Adjust the pH to 5-8 with 10 N NaOH or 1:1 H_2SO_4 as appropriate.

8.3.10 Add enough carbon filtered water to the extractor flask to allow the solvent in the removable sidearm to just begin to drip into the round bottom flask. Record the total volume carbon filtered water that was added on the prep sheet.

8.3.11 Remove the condenser from the rack and rinse the lower joint and lip with a squirt bottle filled with CH_2Cl_2. Place the condenser on the top of the extractor. Turn on the water chiller (set at 10°C) and check the flow indicators.

8.3.12 Turn on the heating mantle so that the indicator light is just brightly lit. Record the starting time on the prep sheet. Check after 15 min to be sure that the solvent in the round bottom flask is boiling, that solvent is dripping from the lip of the condenser, and that the volume of the solvent in the round bottom flask is still about 250 mL. Check all extractor joints for leaks with a Kimwipe.

8.3.13 Allow the extraction to proceed for a minimum of 6 hr.

8.3.14 Turn off the heating mantle and allow the apparatus to cool 30-60 min with the water flowing through the condenser.

8.3.15 The solvent contained in the round bottom flask is the extract. Transfer the extract to a rinsed 400 mL beaker. Rinse the flask with a small amount of CH_2Cl_2. If the volume of solvent is less than about 250 mL, record it on the prep sheet.

8.3.16 Go to part I, step 10 and proceed with the prep.

8.4 Florisil Clean-up for Pesticides

Method Summary: A sample extract in hexane is subjected to a florisil adsorption column clean-up to remove interferences and separate pesticides into discrete fractions. The extract is introduced onto the florisil and eluted into fractions with solvents of varying polarities.

Procedure

8.4.1 Prepare the eluting solvents in 250 mL stoppered graduated cylinders.

Fraction 1: 200 mL 6% diethyl ether in petroleum ether
Fraction 2: 200 mL 15% diethyl ether in petroleum ether
Fraction 3: 200 mL 50% diethyl ether in petroleum ether
Fraction 4: 200 mL 100% diethyl ether in petroleum ether

8.4.2 A column performance standard, OPP CPS, accompanies every set of pesticide florisil columns.

8.4.3 Place a glass wool plug in the bottom of a 20 mm I.D. chromatographic column. Add 20 g of florisil, mesh size 60-100.

8.4.4 Tap the top of the column with a rubber stopper to settle the florisil. Add 1 cm of dried Na_2SO_4 to the top of the column and tap again.

8.4.5 Pre-elute the column with 30-50 mL of petroleum ether. Collect the eluate in a waste beaker. Just prior to the exposure of the Na_2SO_4 to the air, quantitatively transfer 10.0 mL (for industrial samples) of sample extract in toluene onto the top of the column. Continue collecting in a waste beaker.

8.4.6 Just prior to the exposure of the sodium sulfate to the air, add Fraction 1. Collect this eluate in a rinsed 400 mL beaker labeled "Column Cleaned-A".

8.4.7 Just prior to the exposure of the sodium sulfate layer to the air, add Fraction 2. Collect this eluate in the same beaker as above until the solvent volume reaches about 300 mL. Replace beaker with another rinsed beaker labeled "Column Cleaned-B".

8.4.8 Again, just prior to the exposure of the sodium sulfate to the air, add Fraction 3. Continue collecting the eluate in Beaker B. Just prior to the exposure of the sodium sulfate to the air, add Fraction 4. Collect this eluate in another rinsed beaker labeled "Column Cleaned - C."

8.4.9 Pour the eluate from Beaker A into a Kuderna-Danish (K-D) flask. It is not necessary to pass the eluate through sodium sulfate.

8.4.10 Add a boiling chip to the K-D. Attach a 3-ball Snyder column and prewet by adding about 1 mL of petroleum ether to the top.

8.4.11 Place the K-D on a heated water bath set at 95°C so that the receiver tube is partially immersed in hot water and the entire lower rounded surface is bathed in steam. At a proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 5-10 mL, remove the K-D from the water and allow it to cool.

8.4.12 When the flask is cool, add the eluate from Beaker B, add a fresh boiling chip, and repeat step 11. Repeat the procedure with Beaker C.

8.4.13 Add 50 mL of hexane to the top of the Snyder column. Add a fresh boiling chip to the flask and shake the Snyder column to make sure the balls are not stuck.

8.4.14 When the bath has re-heated, return the K-D to the water and concentrate as before, but this time to an apparent volume of 2-5 mL.

8.4.15 After the K-D has completely cooled, rinse the column and the middle flask with a small amount of hexane. Adjust the final volume to equal the volume of extract that was

cleaned. Mark the meniscus, label the vial, and place the extract with the others in the same project, if appropriate.

8.5 Extraction of Solids

NOTE: All glassware must be solvent rinsed before use.

8.5.1 Weigh 30 g of the sample into a 250 mL centrifuge bottle. Add 60 g of dried Na_2SO_4 and stir the mixture well with a steel spatula. The sample should have a grainy texture. If it forms a large clump, add more Na_2SO_4 and note it on the prep sheet.

8.5.2 Add 100 mL of CH_2Cl_2 to all samples.

8.5.3 Add the appropriate surrogate standard using a disposable pipet.

8.5.4 Add the spike, if appropriate, using the procedure described in the previous step.

8.5.5 Sonicate the samples for 1.5 min at an output setting of 10 with the 3/4 in sonicator horn 1/2 in below the surface of the solvent. The sonicator should be in the 1 s pulse mode, with the duty cycle set at 50%. Centrifuge the samples for 3-5 min at 35K RPM.

8.5.6 Decant the solvent layer into a CH_2Cl_2 rinsed 400 mL beaker.

8.5.7 Repeat the extraction twice more using 100 mL aliquots of CH_2Cl_2 each time. Collect these extracts in the same beaker described in step 6.

8.5.8 Record the total volume of the solvent that is recovered on the prep sheet.

8.5.9 Put a plug of glass wool in a funnel and fill 2/3 full with Na_2SO_4. Rinse the funnel and Na_2SO_4 with 30-40 mL of CH_2Cl_2, discard. Pour the extract through the Na_2SO_4 into a 500 mL Kuderna-Danish (K-D) evaporative concentrator. Rinse the beaker then the Na_2SO_4 with small amounts of CH_2Cl_2 into the K-D.

8.5.10 Add a boiling chip to the K-D and attach a 3 ball Snyder to the top. Pre-wet the column by adding about 1 mL of CH_2Cl_2 to the top.

NOTE: The concentration step is critical; losses of target compounds can occur if care is not taken.

8.5.11 Place the K-D in a heated water bath set at 95°C so that the receiver tube is immersed in hot water and the entire lower rounded surface is bathed in steam. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 2-5 mL, remove the K-D from the bath and allow it to completely cool.

8.5.12 If the extract is highly colored or a precipitate forms during concentration, consult a supervisor. See Part III for florisil column cleanup.

8.5.13 After the K-D has cooled, rinse the Snyder column and middle flask with a small amount of CH_2Cl_2. Transfer the extract to a calibrated 15 mL centrifuge tube, rinsing with a small amount of CH_2Cl_2. Be sure to rinse all of the ground glass joints well, as compounds collect on the ground glass.

8.5.14 Carefully concentrate the extract to 2.0 mL under a gentle stream of nitrogen using the N-evap apparatus. Remove from bath and allow to cool.

8.5.15 Add 2 mL of toluene and return to the bath, evaporate the extract to slightly below the 2 mL vortex mark on the tube. Remove the tube from the bath and allow it to cool completely.

8.5.16 Adjust the volume with toluene to 10.0 mL. If the extract is highly colored, forms a precipitate, or stops evaporating, consult a supervisor

8.5.17 Transfer the extract to a labeled 12 mL screw-top vial with a Teflon lined cap, mark the meniscus, and place on a rack with other samples with the same project number, if any.

8.5.18 Complete the prep sheet for the extraction and concentration steps.

8.6 Sulfur Cleanup

8.6.1 Transfer 2.0 mL of the pesticide extract into a 12 mL screw top vial with a Teflon lined cap.

8.6.2 Add 1.0 mL of Tetrabutylammonium (TBA) sulfite reagent and 2.0 mL of 2-propanol to the vial. Cap the vial and shake for at least 1 minute.

8.6.3 If the sample is colorless or the initial color is unchanged and if clear crystals are present, proceed to step 4. However, if there are no crystals present, add dried sodium sulfite in small portions and shake. Repeat until clear crystals are observed.

8.6.4 Add 5 mL of carbon filtered water and shake for at least 1 min. Allow the sample to settle for 5-10 min, then transfer the hexane (top) layer into a cleaned 12 mL vial.

8.6.5 Add a small amount (less than 1 cm) of dried Na_2SO_4 and shake for 1 min. Enough Na_2SO_4 has been added if it remains grainy after shaking.

8.6.6 Transfer the hexane to a 12 mL vial labeled as "Sulfur Cleaned" and store with other samples in the same project.

8.6.7 Fill out a "Column Cleanup Worksheet" for the cleanup.

8.6.8 <u>Preparation of the TBA-sulfite Reagent</u>:

8.6.8.1 Dissolve 3.39 g of tetrabutylammonium hydrogen sulfite in 100 mL of distilled water.

8.6.8.2 To remove impurities, extract this solution 3 times with 20 mL portions of hexane in 500 mL separatory funnel. Discard the hexane extracts.

8.6.8.3 Add 25 g Na_2SO_4 sulfite to the water solution.

8.6.8.4 Store the resulting solution, which is saturated with Na_2So_4, in am amber bottle with a Teflon lined screw cap.

8.6.8.5 This solution can be store for approximately 1 mo at room temperature.

8.6.9 <u>Testing Extracts for Sulfur:</u>

8.6.9.1 Transfer a small aliquot of extract, $\leq$ 0.5 mL, to a clean vial.

8.6.9.2 Add 1 to 3 drops of mercury to the vial and seal.

8.6.9.3 Shake the vial for 15-30 s. Let the vial stand for 5-10 min.

8.6.9.4 Any discoloration or tarnishing of the mercury indicates that the sample contains sulfur.

8.6.9.5 Prepare a control sample also. Pipet 0.5 mL of hexane to a clean vial and add 1 to 3 drops of mercury. There should be no physical change in the mercury droplet.

8.6.10 To Dispose of Mercury Contaminated Waste:

8.6.10.1 Label vials as mercury waste and dispose of in the inorganic standards waste drum.

9. Analysis

9.1 Calibration of Instrument

Calibration standards are run at 5 levels and a calibration curve generated for each compound. Most compounds are linear in the range of 0.05-2.5 ug/mL. Correlation coefficients $\geq$0.995 are expected for each analyte. External standard tables are used to quantify samples.

Run a mid level check standard after every 10th injection and at the end of the sequence. Precede each check standard with a solvent blank. Response factors should not exceed $\pm$15 % of the regression curve.

9.2 GC Conditions

Second column confirmation is required for all samples that contain any target compounds. Dissimilar columns may be operated simultaneously. The following conditions are suggested.

injection Port	220°C
detection	250°C
injection volume	2 uL
initial temperature	50°C
hold	3 min
ramp rate	12°C/min
final temperature	240°C
hold	20 min
carrier gas (He)	5 mL/min
auxiliary gas (N_2)	15-20 mL/min
air	150 mL/min
H_2	75 mL/min

10. QC/QA requirements

10.1 QC samples

10.1.1 For organophosphorous analyses which are RMAL standard products, 2 laboratory control samples are generated for every 20 samples and a spike control sample with every batch prepped.

10.1.2 Project-specific QC such as matrix spikes and duplicates may be requested.

10.2 Acceptance criteria.

AQUEOUS MATRIX

Analyte(s)	Spike Conc.	Accuracy Control Limits	Precision Control Limits
Diazinon	10 ug/L	20-160	20%
Methyl Parathion	10 ug/L	20-160	20%
Malathion	10 ug/L	20-160	20%
Ethion	10 ug/L	20-160	20%

SOLID MATRIX

Analyte(s)	Spike Conc.	Accuracy Control Limits	Precision Control Limits
Diazinon	330 ug/kg	20-160	20%
Methyl Parathion	330 ug/kg	20-160	20%
Malathion	330 ug/kg	20-160	20%
Ethion	330 ug/kg	20-160	20%

80% of the spike analytes must fall within control limits.

10.3 For corrective action see the current QC manual.

11. Calculations

11.1 Concentrations of analytes may be read directly from the external standard table. These values are in ug/mL and from them final sample concentrations may be calculated.

11.1.1 For waters:

$$\frac{ug}{mL} \text{ of analyte} \times \frac{\text{final vol (mL)}}{\text{liters extracted}} \times \text{dilution factor} = \frac{ug}{L}$$

11.1.2 For soils:

$$\frac{ug}{mL} \text{ of analyte} \times \frac{\text{final vol (mL)}}{\text{kg extracted}} \times \text{dilution factor} = \frac{ug}{kg}$$

11.2 Reporting units are ug/L for liquids and ug/kg for solids.

11.3 Reporting limits are based on the lowest standard run. See Section 1.2.

11.4 Results are reported to 2 significant figures unless the client specifically requests otherwise. Results below detection limit are reported as not detected (ND).

11.5 LIMS Data Entry

12. References

12.1 Method 614, EMSL - Cincinnati, January, 1982.

12.2 USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460, November, 1986, SW-846, Third Edition.

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Industrial Raw Data Packages

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1. Purpose

   This Standard Operating Procedure (SOP) defines the deliverables and documentation included in a raw data package for examination by an experienced reviewer.

2. Policies

   Enseco-RMAL provides raw data packages upon request for the purpose of sample data validation. If samples are reanalyzed due to interference and matrix effect is proven, both sets of analyses data are included in the data package.

Prepared by:	Date:
Julie Kramer	January 8, 1991
Management Approval: Gary Tsai	Date: 3-19-91
QA Officer Approval: Jan Redenbarger	Date: 3-19-91

3. Safety Issues

None.

4. Procedure

The following sections detail the deliverables and documentation required for a raw data package by department.

4.1 GC/MS Standard Short Form Raw Data Package

4.1.1 Copy of all samples, MS, MSD, and DU raw data for each reported analysis and/or reanalysis. Raw data includes sample chromatograms (RIC), quantitation summary, diagnostic report, spectra, TIC report, and library searches when applicable.

4.1.2 Copy of summary from tuning raw data for each day samples MS, MSD, and DU were analyzed and for each instrument. Raw data includes bar graph spectra and mass listing.

4.1.3 Copy of initial calibration summary sheet, listing all response factors, %RSD, etc.

4.1.4 Copy of daily continuing calibration summary for each day samples were analyzed and for each instrument.

4.1.5 Copy of sample preparation logs (if applicable) and analysis log book pages.

4.2 GC/MS Extended Long Form Raw Data Package

4.2.1 Copy of all samples and quality control samples (MS, MSD, DU, SCS, DCS) raw data for each reported analysis and/or reanalysis. Raw data includes sample chromatograms (RIC), summary sheet, quantitation summary, diagnostic report, spectra, TIC report, and library searches (when applicable).

4.2.2 Copy of tuning raw data for each day samples and QC (MS, MSD, DU, SCS, DCS) were analyzed and for each instrument. Raw data includes bar graph spectra and mass listing. Tuning raw data for associated initial calibrations are included.

4.2.3 Copy of initial calibration raw data for each instrument. Raw data includes initial calibration summary from listing response factors and %RSD, chromatograms, quantitation summary, and diagnostic report.

4.2.4 Copy of daily continuing calibration raw data for each day samples and QC (MS, MSD, DU, SCS, DCS) are analyzed and for each instrument. Raw data includes continuing calibration summary form, chromatograms, quantitation summary, and diagnostic report.

4.2.5 Copy of sample preparation logs (if applicable) and analysis log book pages for QC and samples.

4.3 GC Standard Short Form Raw Data Package

4.3.1 Copy of all samples, MS, MSD, and DU raw data for each reported analysis and/or reanalysis. Raw data includes analysis report and chromatogram for each column.

4.3.2 Copy of summary from initial calibration, listing all response factors, and %RSD.

4.3.3 Copy of bracketing daily continuing calibration standards for each reported analysis. Raw data includes analysis report and chromatogram for each column.

4.3.4 Copy of sample preparation logs (if applicable) and analysis log book pages.

4.4 GC Standard Extended Long Form Raw Data Package

4.4.1 Copy of all sample and QC (MS, MSD, DU, SCS, DCS) raw data for each reported analysis and/or reanalysis. Raw data includes analysis report and chromatogram for each column.

4.4.2 Copy of initial calibration raw data for each reported analysis. Raw data includes initial standards and summary.

4.4.3 Copy of all bracketing continuing calibration standards for each reported analysis. Raw data includes analysis report and chromatogram for each column.

4.4.4 Copy of sample preparation logs (if applicable) and analysis log book pages.

4.5 Metals

4.5.1 Copy of all reported sample raw data for ICP, GFAA, and/or CVAA (Mercury) analyses. Raw data includes ICP computer printouts as well as GFAA and CVAA benchsheets for all samples and matrix specific QC (MS, MSD, DU).

4.5.2 Copy of initial calibration raw data for ICP, GFAA, and/or CVAA analyses. Raw data includes ICP computer printouts as well as GFAA and CVAA benchsheets for ICV, ICS (ICP only), ICB, DCS, and preparation blank analyses.

4.5.3 Copy of all continuing calibration raw data for ICP, GFAA, and/or CVAA analyses. Raw data includes ICP computer printouts as well as GFAA and CVAA benchsheets for all CCV, CCB analyses.

4.5.4 Copy of sample preparation and leachate (TCLP) sheets for all samples (if applicable).

4.6 Wet Chemistry

4.6.1 Copy of all sample preparation and leachate (DI) documentation when applicable.

4.6.2 Copy of all standard curve and correlation coefficients for each day samples are analyzed.

4.6.3 Copy of all benchsheets for associated DCS and method blank analyses.

4.6.4 Copy of analysis benchsheets for all samples and matrix specific QC (spike, duplicate) samples, for each analytical parameter requested.

4.6.5 Copy of all instrument chromatograms and readouts.

5. Responsibilities

It is the responsibility of each departmental data review and data control group to ensure that the raw data package is complete and organized at the time of submission to the client. The data package shall be organized in the format listed below.

5.1 Raw Data Format

5.1.1 The raw data package shall be put together in order of department and analytical test. The order shall be as follows: GC/MS (VOA, Semivoa), GC (VOA, Semivoa), Metals (ICP, GFAA, CVAA), and Wet Chemistry (no particular order of tests). Divider sheets which separate sample, calibration, project specific QC, DCS, SCS, Method Blank, and extraction/analysis logbook pages shall be inserted into the appropriate place in the data package.

5.1.2 Table of Contents

The completed raw data package shall be paginated and a table of contents shall be submitted listing the paginated page numbers from the divider sheets.

5.1.3 Other Documentation

A signed copy of the sample Chain-of-Custody shall be included in the raw data package.

A copy of the sample receipt checklist generated at the time of log-in shall be included in the raw data package.

6. Comments

The raw data package provides sufficient documentation to allow an experienced reviewer to independently reconstruct the reported results. In some situations, additional documentation may be required.

7. Definitions

Refer to the ENSECO QAPP for definitions of DSC, SCS, etc.

ATTACHMENTS

Item 1

Standard Industrial Organic Raw Data Packages

SHORT FORM pkg.*

GC/MS

- -sample data
- -BFB or DFTPP
- -initial calibration summary
- -continuing calibration summary
- -DCS and SCS summaries
- -analysis log pages, IS check summary
- -prep sheets (if applicable)

GC

- -sample data
- -initial calibration summary
- -bracketing cont. calibration standards (analysis report and plot for each column)
- -analysis log pages
- -prep sheets (if applicable)

LONG FORM pkg.*

GC/MS

- -sample data
- -DCS
- -SCS
- -BFB or DFTPP (for both samples and Enseco QC)
- -initial calibration (Full package for samples summary for GC)
- -continuing calibration (for both samples and Enseco QC)
- -analysis log pages, IS check summary
- -prep sheets (if applicable)

GC

- -sample data
- -DCS
- -SCS
- -initial calibration
- -bracketing cont. calibration standards (analysis report and plot for each column and for both samples and Enseco QC.)
- -analysis log pages
- -prep sheets (if applicable)

* Unless summary is specified, or other parts specified, all raw data is needed.

** Report Writing provides Full Package of standards for initial calibration.

ATTACHMENTS
(cont)

Item 2

Raw Data

VOA
- Extraction Logs (if applicable)
- Analysis Logs-for samples only
- Sample Data-in order numerically
- Specific QC (MS/SD)
- Tuning Data-by instrument by date
- Initial calibration-5 pt. summary only
- Continuing calibration-summary + chromatograms
- DCS
  SCS/Blank

BNA
- Same as VOA with exception of extraction logs

  Note: Data should be put in order of instrument + date

GC
- Extraction logs/analysis logs
- Sample data
- Specific QC (MS/SD)
- Standards
  - Initial curve
  - Cont. check standards (must bracket samples)
  - DCS
  - SCS

ATTACHMENTS
(cont)

Format

1. Prep sheet, reprep sheets, reprep request sheets
2. Element Order
   a. As (FAA)
   b. Pb (FAA)
   c. Hg (CVAA)
   d. Se (FAA)
   e. Ti (FAA)
   f. Miscellaneous (FAA)
   g. ICP
3. Matrix Order
   a. Soils/aqueous total
   b. Aqueous dissolved
   c. TCLP
   e. FAA
4. All elements will be in order of analyzed date.
5. All reanalysis request sheets will be in front of element needing reanalysis.

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Reduced by: RE Date: 5-7-92 Initial Calibration Report
iewed by: _____ Date: _____

Project: OPP04_3, Channel: 52, Analysis: D22APR92, Node: DENCR3
Analyst: A.STEYERT, Calibration File: OPP0422, MCRPT Method: OPPA

Calibration curve type: Linear NOT forced through zero

Sample	Level	ID	Description
2	L5	8140	CSA 0.5% #5940 T=ST
3	L4	8140	CSA 0.4% #5940 T=ST
4	L3	8140	CSA 0.2% #5940 T=ST
5	L2	8140	CSA 0.1% #5940 T=ST
6	L1	8140	CSA 0.05% #5940 T=ST

%RSD Measured	Maximum	Compound
11.1	20.0	DICHLORVOS
17.2	20.0	PHOSDRIN
6.8	20.0	CHLORMEFOS
4.3	20.0	ETHOPROP
65.0	20.0*	NALED ←
6.2	20.0	SULFOTEPP
6.4	20.0	PHORATE
6.8	20.0	DEMETON (O,S)
7.4	20.0	DIMETHOATE
3.4	20.0	DIAZINON
2.8	20.0	DISYSTON
8.5	20.0	METHYL PARATHION
3.9	20.0	RONNEL
4.4	20.0	MALATHION
4.2	20.0	FENTHION
5.0	20.0	CHLORPYRIFOS
5.0	20.0	ETHYL PARATHION
6.7	20.0	TRICHLORONATE
6.1	20.0	ETHYL PIRIMIPHOS
10.4	20.0	MERPHOS
26.4	20.0*	TETRACHLORVINPHOS
6.3	20.0	PROTHIOPHOS
2.9	20.0	DEF
17.9	20.0	FENSULFOTHION
4.0	20.0	ETHION
2.8	20.0	SULPROFOS
13.0	20.0	METHYL AZINPHOS
10.0	20.0	COUMAPHOS

Samp 2 Meas True %Diff	Samp 3 Meas True %Diff	Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
DICHLORVOS							
0.2604	0.2220	0.1050	0.04465	0.02263			
0.2500	0.2000	0.1000	0.05000	0.02500			
4.2	11.0	5.0	-10.7	-9.5			
PHOSDRIN							
0.3898	0.3549	0.1613	0.06694	0.03037			
0.3750	0.3000	0.1500	0.07500	0.03750			
3.9	18.3	7.5	-10.7	-19.0			
CHLORMEFOS							
0.4758	0.4028	0.1936	0.09746	0.05493			
0.5000	0.4000	0.2000	0.1000	0.05000			
-4.8	0.7	-3.2	-2.5	9.9			
ETHOPROP							
0.2362	0.2090	0.09943	0.04979	0.02551			
0.2500	0.2000	0.1000	0.05000	0.02500			
-5.5	4.5	-0.6	-0.4	2.0			
NALED							
882	1.584	0.4991	0.1639	0.03199			
250	1.000	0.5000	0.2500	0.1250			
50.6	58.4	-0.2	-34.4	-74.4			
SULFOTEPP							
0.2416	0.2117	0.1003	0.04648	0.02607			
0.2500	0.2000	0.1000	0.05000	0.02500			
-3.4	5.8	0.3	-7.0	4.3			
PHORATE							
0.2412	0.2188	0.09639	0.04853	0.02518			
0.2500	0.2000	0.1000	0.05000	0.02500			
-3.5	9.4	-3.6	-2.9	0.7			
DEMETON (O,S)							
0.2468	0.2178	0.1003	0.04620	0.02491			
0.2500	0.2000	0.1000	0.05000	0.02500			
-1.3	8.9	0.3	-7.6	-0.4			
DIMETHOATE							
0.2626	0.2414	0.1028	0.04526	0.02025			
0.2500	0.2000	0.1000	0.05000	0.02500			
5.0	20.7	2.8	-9.5	-19.0			
DIAZINON							
0.2394	0.2020	0.09992	0.04969	0.02599			
2500	0.2000	0.1000	0.05000	0.02500			
-4.2	1.0	-0.1	-0.6	3.9			

Samp 2 Meas True %Diff	Samp 3 Meas True %Diff	Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
DISYSTON							
0.2428	0.2022	0.1031	0.04903	0.02514			
0.2500	0.2000	0.1000	0.05000	0.02500			
-2.9	1.1	3.1	-1.9	0.6			
METHYL PARATHION							
0.2539	0.2106	0.1015	0.05223	0.02180			
0.2500	0.2000	0.1000	0.05000	0.02500			
1.6	5.3	1.5	4.5	-12.8			
RONNEL							
0.2437	0.2014	0.1050	0.05020	0.02410			
0.2500	0.2000	0.1000	0.05000	0.02500			
-2.5	0.7	5.0	0.4	-3.6			
MALATHION							
0.6465	0.5185	0.2521	0.1194	0.06031			
0.6250	0.5000	0.2500	0.1250	0.06250			
3.4	3.7	0.8	-4.5	-3.5			
FENTHION							
^ 2548	0.2083	0.1007	0.04936	0.02362			
2500	0.2000	0.1000	0.05000	0.02500			
1.9	4.2	0.7	-1.3	-5.5			
CHLORPYRIFOS							
0.4775	0.3988	0.1980	0.1073	0.04926			
0.5000	0.4000	0.2000	0.1000	0.05000			
-4.5	-0.3	-1.0	7.3	-1.5			
ETHYL PARATHION							
0.4775	0.3988	0.1980	0.1073	0.04926			
0.5000	0.4000	0.2000	0.1000	0.05000			
-4.5	-0.3	-1.0	7.3	-1.5			
TRICHLORONATE							
0.2368	0.1997	0.09543	0.05464	0.02518			
0.2500	0.2000	0.1000	0.05000	0.02500			
-5.3	-0.1	-4.6	9.3	0.7			
ETHYL PIRIMIPHOS							
0.4882	0.3940	0.1935	0.09771	0.05470			
0.5000	0.4000	0.2000	0.1000	0.05000			
-2.4	-1.5	-3.2	-2.3	9.4			
MERPHOS							
0.6796	0.5392	0.2551	0.1162	0.05527			
5250	0.5000	0.2500	0.1250	0.06250			
8.7	7.8	2.0	-7.0	-11.6			

	Samp 2 Meas True %Diff	Samp 3 Meas True %Diff	Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
TETRACHLORVINPHOS								
	0.7477	0.6023	0.2664	0.1068	0.04243			
	0.6250	0.5000	0.2500	0.1250	0.06250			
	19.6	20.5	6.6	-14.5	-32.1			
PROTHIOPHOS								
	0.2408	0.2000	0.09695	0.04861	0.02738			
	0.2500	0.2000	0.1000	0.05000	0.02500			
	-3.7	0.0	-3.0	-2.8	9.5			
DEF								
	0.6256	0.5184	0.2468	0.1212	0.06286			
	0.6250	0.5000	0.2500	0.1250	0.06250			
	0.1	3.7	-1.3	-3.1	0.6			
FENSULFOTHION								
	1.434	1.151	0.5071	0.2199	0.1011			
	1.250	1.000	0.5000	0.2500	0.1250			
	14.7	15.1	1.4	-12.0	-19.1			
ETHION								
	2463	0.2010	0.09837	0.04845	0.02643			
	2500	0.2000	0.1000	0.05000	0.02500			
	-1.5	0.5	-1.6	-3.1	5.7			
SULPROFOS								
	0.2502	0.1998	0.1000	0.04827	0.02586			
	0.2500	0.2000	0.1000	0.05000	0.02500			
	0.1	-0.1	0.0	-3.5	3.4			
METHYL AZINPHOS								
	1.321	1.118	0.5239	0.2366	0.1039			
	1.250	1.000	0.5000	0.2500	0.1250			
	5.7	11.8	4.8	-5.4	-16.9			
COUMAPHOS								
	0.6579	0.5421	0.2605	0.1184	0.05464			
	0.6250	0.5000	0.2500	0.1250	0.06250			
	5.3	8.4	4.2	-5.3	-12.6			

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Reduced by: RE Date: 5-7-92 Initial Calibration Report
I iewed by: _____ Date: _____

Project: OPP04_3, Channel: 51, Analysis: D22APR92, Node: DENCR3
Analyst: A.STEYERT, Calibration File: OPP0422, MCRPT Method: OPPA

Calibration curve type: Linear NOT forced through zero

Sample	Level	ID	Description				
2	L5	8140	CSA	0.5%	#5940	T=ST	
3	L4	8140	CSA	0.4%	#5940	T=ST	
4	L3	8140	CSA	0.2%	#5940	T=ST	
5	L2	8140	CSA	0.1%	#5940	T=ST	
6	L1	8140	CSA	0.05%	#5940	T=ST	

Measured	%RSD Maximum	Compound
14.4	20.0	DICHLORVOS
21.8	20.0*	PHOSDRIN
20.0	20.0*	CHLORMEFOS
5.2	20.0	ETHOPROP
56.6	20.0*	NALED
5.2	20.0	SULFOTEPP
5.1	20.0	PHORATE
8.8	20.0*	DIMETHOATE
3.4	20.0	DEMETON (O,S)
6.4	20.0	DIAZINON
6.4	20.0	DISYSTON
8.7	20.0	METHYL PARATHION
7.1	20.0	RONNEL
11.8	20.0	MALATHION
3.1	20.0	FENTHION
9.7	20.0	ETHYL PARATHION
5.2	20.0	CHLORPYRIFOS
5.0	20.0	TRICHLORONATE
3.0	20.0	ETHYL PIRIMIPHOS
8.8	20.0	MERPHOS
22.8	20.0*	TETRACHLORVINPHOS
4.1	20.0	PROTHIOPHOS
1.8	20.0	DEF
23.8	20.0*	FENSULFOTHION
3.1	20.0	ETHION
4.9	20.0	SULPROFOS
21.2	20.0*	METHYL AZINPHOS
14.1	20.0	COUMAPHOS

Samp 2 Meas True %Diff	Samp 3 Meas True %Diff	Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
DICHLORVOS							
0.2685	0.2217	0.1083	0.04507	0.02082			
0.2500	0.2000	0.1000	0.05000	0.02500			
7.4	10.8	8.3	-9.9	-16.7			
PHOSDRIN							
0.3626	0.3242	0.1880	0.07210	0.02765			
0.3750	0.3000	0.1500	0.07500	0.03750			
-3.3	8.1	25.4	-3.9	-26.3			
CHLORMEFOS							
0.3989	0.3576	0.2072	0.1014	0.06289			
0.5000	0.4000	0.2000	0.1000	0.05000			
-20.2	-10.6	3.6	1.4	25.8			
ETHOPROP							
0.2455	0.2070	0.1003	0.04672	0.02615			
0.2500	0.2000	0.1000	0.05000	0.02500			
-1.8	3.5	0.3	-6.6	4.6			
NALED							
849	1.480	0.5016	0.1646	0.04743			
250	1.000	0.5000	0.2500	0.1250			
47.9	48.0	0.3	-34.2	-62.1			
SULFOTEPP							
0.2448	0.2080	0.1014	0.04660	0.02587			
0.2500	0.2000	0.1000	0.05000	0.02500			
-2.1	4.0	1.4	-6.8	3.5			
PHORATE							
0.2452	0.2076	0.1010	0.04670	0.02592			
0.2500	0.2000	0.1000	0.05000	0.02500			
-1.9	3.8	1.0	-6.6	3.7			
DIMETHOATE							
0.2980	0.2505	0.1069	0.04097	0.01669			
0.2500	0.2000	0.1000	0.05000	0.02500			
19.2	25.3	6.9	-18.1	-33.2			
DEMETON (O,S)							
0.2361	0.2036	0.09569	0.04802	0.02801			
0.2500	0.2000	0.1000	0.05000	0.02500			
-5.6	1.8	-4.3	-4.0	12.0			
DIAZINON							
0.4867	0.4143	0.2012	0.09214	0.05318			
5000	0.4000	0.2000	0.1000	0.05000			
-2.7	3.6	0.6	-7.9	6.4			

	Samp 2 Meas True %Diff	Samp 3 Meas True %Diff	Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
DISYSTON								
	0.4867	0.4143	0.2012	0.09214	0.05318			
	0.5000	0.4000	0.2000	0.1000	0.05000			
	-2.7	3.6	0.6	-7.9	6.4			
METHYL PARATHION								
	0.2422	0.2043	0.1021	0.04466	0.02740			
	0.2500	0.2000	0.1000	0.05000	0.02500			
	-3.1	2.2	2.1	-10.7	9.6			
RONNEL								
	0.2465	0.2062	0.09758	0.04612	0.02712			
	0.2500	0.2000	0.1000	0.05000	0.02500			
	-1.4	3.1	-2.4	-7.8	8.5			
MALATHION								
	0.6709	0.5533	0.2592	0.1134	0.05477			
	0.6250	0.5000	0.2500	0.1250	0.06250			
	7.3	10.7	3.7	-9.3	-12.4			
FENTHION								
	.2513	0.2072	0.1012	0.04848	0.02443			
	.2500	0.2000	0.1000	0.05000	0.02500			
	0.5	3.6	1.2	-3.0	-2.3			
ETHYL PARATHION								
	0.2606	0.2158	0.1055	0.04701	0.02209			
	0.2500	0.2000	0.1000	0.05000	0.02500			
	4.2	7.9	5.5	-6.0	-11.7			
CHLORPYRIFOS								
	0.2371	0.1957	0.09914	0.05061	0.02674			
	0.2500	0.2000	0.1000	0.05000	0.02500			
	-5.2	-2.2	-0.9	1.2	7.0			
TRICHLORONATE								
	0.2457	0.2026	0.1004	0.04703	0.02647			
	0.2500	0.2000	0.1000	0.05000	0.02500			
	-1.7	1.3	0.4	-5.9	5.9			
ETHYL PIRIMIPHOS								
	0.4985	0.4119	0.2034	0.09605	0.04978			
	0.5000	0.4000	0.2000	0.1000	0.05000			
	-0.3	3.0	1.7	-3.9	-0.4			
MERPHOS								
	0.6625	0.5358	0.2578	0.1131	0.05829			
	6250	0.5000	0.2500	0.1250	0.06250			
	6.0	7.2	3.1	-9.5	-6.7			

	Samp 2 Meas True %Diff	Samp 3 Meas True %Diff	Samp 4 Meas True %Diff	Samp 5 Meas True %Diff	Samp 6 Meas True %Diff	Meas True %Diff	Meas True %Diff	Meas True %Diff
TETRACHLORVINPHOS								
	0.7370	0.5952	0.2610	0.1012	0.04855			
	0.6250	0.5000	0.2500	0.1250	0.06250			
	17.9	19.0	4.4	-19.0	-22.3			
PROTHIOPHOS								
	0.2557	0.2048	0.1015	0.05002	0.02346			
	0.2500	0.2000	0.1000	0.05000	0.02500			
	2.3	2.4	1.5	0.0	-6.2			
DEF								
	0.6109	0.5046	0.2539	0.1239	0.06292			
	0.6250	0.5000	0.2500	0.1250	0.06250			
	-2.3	0.9	1.6	-0.9	0.7			
FENSULFOTHION								
	1.485	1.201	0.5181	0.2044	0.09462			
	1.250	1.000	0.5000	0.2500	0.1250			
	18.8	20.1	3.6	-18.3	-24.3			
ETHION								
	2460	0.2043	0.1029	0.04814	0.02508			
	2500	0.2000	0.1000	0.05000	0.02500			
	-1.6	2.1	2.9	-3.7	0.3			
SULPROFOS								
	0.2411	0.2043	0.1065	0.04866	0.02441			
	0.2500	0.2000	0.1000	0.05000	0.02500			
	-3.6	2.1	6.5	-2.7	-2.4			
METHYL AZINPHOS								
	1.405	1.188	0.5352	0.2153	0.09455			
	1.250	1.000	0.5000	0.2500	0.1250			
	12.4	18.8	7.0	-13.9	-24.4			
COUMAPHOS								
	0.6919	0.5592	0.2555	0.1147	0.05217			
	0.6250	0.5000	0.2500	0.1250	0.06250			
	10.7	11.8	2.2	-8.2	-16.5			

Injection Report

Acquired on 22-APR-1992 at 15:36

RMAL Chromatography System on DENCR3

Analyst Name : A.STEYERT
Lims Id : 2 UL INJECT
Comment : D-5890_A, DUAL FPD, RTX-1/RTX-5, 0.25uM, 30 M, 0.25 MM ID
Method Title : OPP 8140
Sample Name : 8140 CSA 0.5% #5940 T=ST
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 2

RE
5-7-92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
1	5.724	0.000	1936	8112	0.00000		10.7
2	5.929	0.000	9373	23411	0.00000		5.9
3	6.142	0.000	2035	7303	0.00000		5.3
4	6.311	0.000	1026	3165	0.00000		5.3
5	6.430	6.430	665615	1062944	0.26847	DICHLORVOS	2.1A
6	6.747	0.000	25020	59354	0.00000		4.3A
7	7.271	0.000	19357	34754	0.00000		3.2A
8	7.476	0.000	7421	33572	0.00000		5.9
9	7.796	7.796	757073	2462644	0.36259	PHOSDRIN	5.3
10	7.929	7.929	755291	1421117	0.39892	CHLORMEFOS	3.2A
11	8.667	0.000	5643	15737	0.00000		3.2A
12	8.924	0.000	2947	15139	0.00000		4.3A
13	9.209	0.000	7314	28429	0.00000		3.7
14	9.573	0.000	89582	194388	0.00000		3.2A
15	9.804	9.804	318672	699859	0.24547	ETHOPROP	3.2A
16	10.053	10.053	211227	498425	1.34934	NALED	3.2A
17	10.427	10.418	566355	1293161	0.24483	SULFOTEPP	3.2A
18	10.604	10.604	257664	625388	0.24518	PHORATE	3.2A
19	10.764	10.764	171683	583508	0.29797	DIMETHOATE	4.3
20	10.978	10.978	162579	505010	0.23608	DEMETON (O,S)	3.7
21	11.680	0.000	1987	16756	0.00000		6.9
C1		12.520	264252	1271051	0.48670	DIAZINON/DISYSTON	
22	12.524	12.520	264252	1271051	0.48670	- C1	4.8
23	14.133	14.133	161311	691956	0.24218	METHYL PARATHION	4.8
24	14.924	0.000	1463	21708	0.00000		18.7
25	15.351	15.351	135031	640408	0.24646	RONNEL	5.3
26	16.720	16.720	228325	1089425	0.67088	MALATHION	5.3
27	17.040	17.040	132484	664515	0.25133	FENTHION	5.9
28	17.298	17.307	132876	662515	0.26055	ETHYL PARATHION	5.9
29	17.440	17.449	114138	585582	0.23709	CHLORPYRIFOS	5.9
30	17.796	0.000	2883	17991	0.00000		10.1
31	18.560	18.569	102834	553222	0.24575	TRICHLORONATE	5.9
32	19.404	19.404	228760	1162366	0.49850	ETHYL PIRIMIPHOS	5.3

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
33	19.733	0.000	1569	10590	0.00000		8.0
34	20.516	20.516	133587	701598	0.66247	MERPHOS	5.9
35	21.280	0.000	1841	9576	0.00000		6.9
36	21.600	0.000	900	3220	0.00000		3.2
37	21.813	0.000	7690	41891	0.00000		6.4
38	22.089	22.089	138626	802786	0.73704	TETRACHLORVINPHOS	5.9
39	22.569	0.000	4590	33089	0.00000		6.9
40	23.831	23.831	82834	488910	0.25571	PROTHIOPHOS	5.9
41	24.489	24.489	293819	1695615	0.61088	DEF	6.4
42	26.338	26.347	280836	2149866	1.48539	FENSULFOTHION	6.9
43	27.304	0.000	4924	28349	0.00000		6.4
44	28.062	28.062	211671	972268	0.24598	ETHION	4.8
45	28.578	28.578	135277	561351	0.24112	SULPROFOS	4.8
46	29.680	0.000	1082	3245	0.00000		4.8
47	30.036	30.036	93773	244232		TRIPHENYLPHOSPHATE	4.3A
48	31.422	31.413	754408	1868314	1.40539	METHYL AZINPHOS	3.2A
49	32.142	0.000	867	1208	0.00000		2.1A
50	32.471	0.000	859	2034	0.00000		3.2A
51	32.996	32.996	371037	873778	0.69187	COUMAPHOS	4.3A
52	33.724	0.000	810	4184	0.00000		5.9
53	34.924	0.000	1052	2579	0.00000		4.8

Totals			
Unknowns	0	0	N/A
Quantified	8066209	27451590	13.62412
Grand Total	8066209	27451590	13.62412

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... OPP
Run sequence..... VER0422
Calibration..... OPP0422
Internal standard calibration using area
Calibration last modified on 23-APR-1992 at 08:11

Uncalibrated peaks use user factor (0.0000)

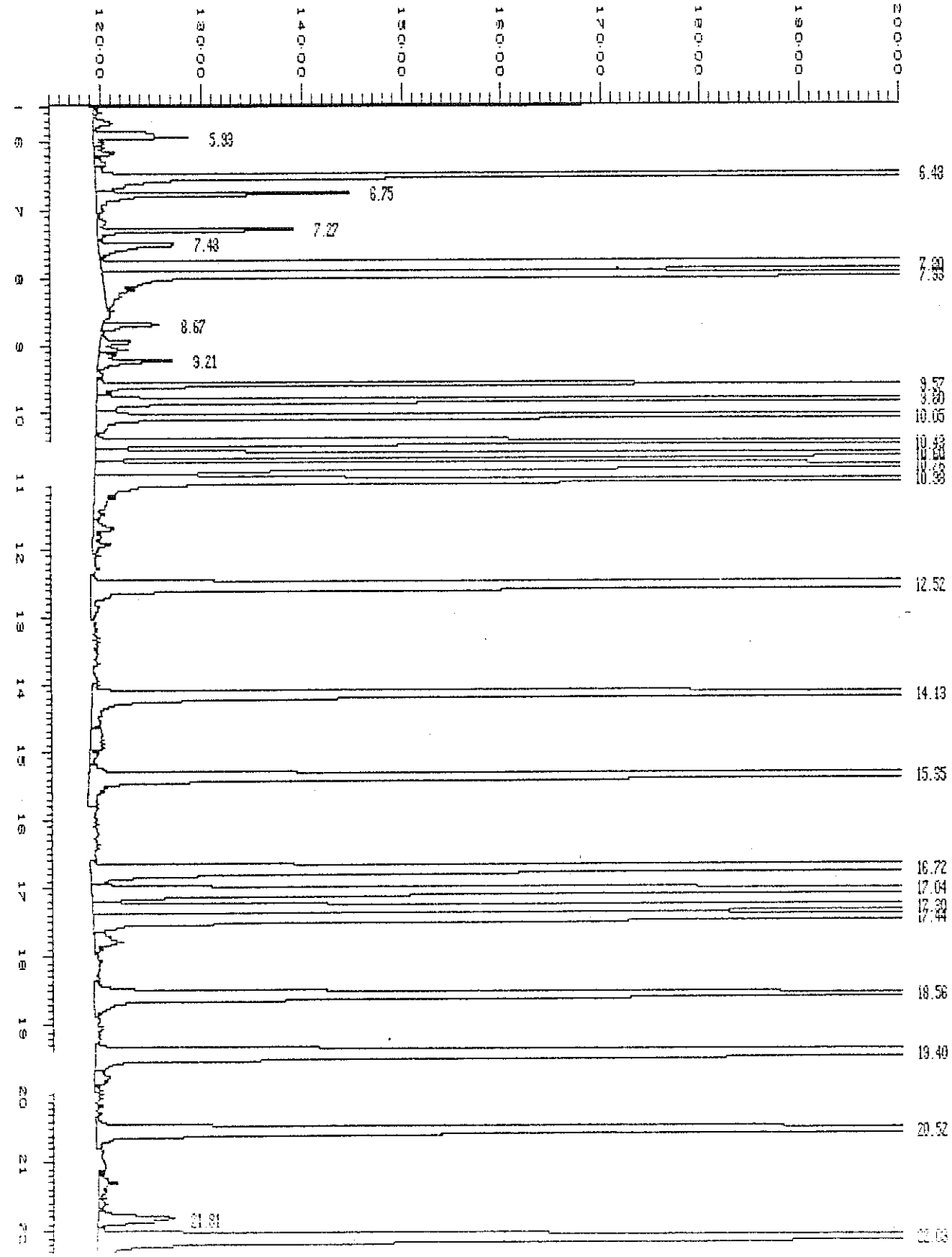
RMAL Chromatography System on DENCR3

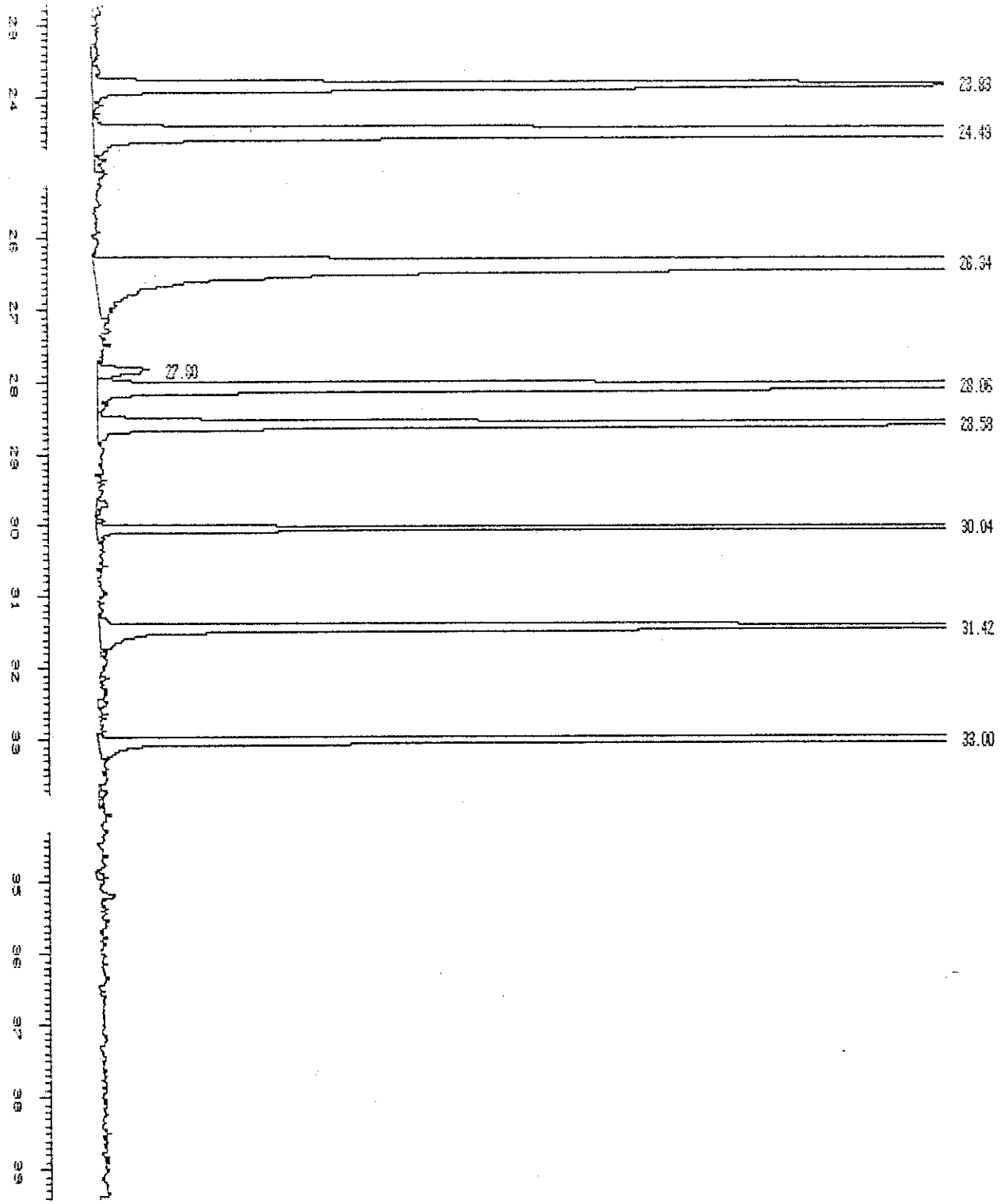
[OPP84_3] S1 D22APR92,2,1

S1-- CSA 0.5% #5940 T=ST. Amount : 1.000.

Acquired on 22-APR-1992 at 15:36
Reported on 22-APR-1992 at 16:23
Box 1 (of 1)

RE
5-7-92





51
D22APR92,2

RE
5-7-92

Injection Report

Acquired on 22-APR-1992 at 15:36

RMAL Chromatography System on DENCR3

Analyst Name : A.STEYERT
Lims Id : 2 UL INJECT
Comment : D-5890_A, DUAL FPD, RTX-1/RTX-5, 0.25uM, 30 M, 0.25 MM ID
Method Title : OPP 8140
Sample Name : 8140 CSA 0.5% #5940 T=ST
Sample Id :
Sample Type : Standard Amount=1.000000
Bottle No : 2

AE
5-7-92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL/L/kg	Peak name	Width
1	6.773	0.000	1380	3157	0.00000		3.2A
2	6.951	0.000	2224	5866	0.00000		2.1A
3	7.111	0.000	3536	6592	0.00000		3.2A
4	7.230	0.000	1890	4948	0.00000		3.2A
5	7.511	0.000	824	4720	0.00000		5.9
6	7.689	7.680	389544	687869	0.26040	DICHLORVOS	2.1A
7	8.000	0.000	11635	31397	0.00000		3.2A
8	8.658	0.000	6970	21755	0.00000		3.2A
9	9.013	0.000	6646	26941	0.00000		4.3A
10	9.431	9.422	753248	2008245	0.38979	PHOSDRIN	3.2A
11	9.636	9.636	353893	832774	0.47582	CHLORMEFOS	3.2A
12	10.827	0.000	2781	9189	0.00000		3.2A
13	11.476	0.000	1212	7018	0.00000		9.6
14	11.778	0.000	3724	17319	0.00000		3.7
15	12.284	0.000	28589	95288	0.00000		4.3A
16	12.747	12.747	95336	332545	0.23615	ETHOPROP	4.3A
17	13.387	13.378	84467	316385	1.88236	NALED	4.3
18	13.938	13.938	174033	649131	0.24156	SULFOTEPP	4.3
19	14.249	14.249	74615	315531	0.24117	PHORATE	4.8
20	14.764	0.000	513	2558	0.00000		9.1
21	15.102	15.102	50089	225883	0.24681	DEMETON (O,S)	4.8
22	15.280	15.289	53982	317451	0.26260	DIMETHOATE	5.9
23	16.444	0.000	909	3420	0.00000		5.3
24	17.573	17.564	64192	308325	0.23942	DIAZINON	5.3
25	17.858	17.849	64885	334032	0.24279	DISYSTON	5.3
26	20.951	20.942	58022	351415	0.25393	METHYL PARATHION	6.4
27	21.564	0.000	1384	6432	0.00000		2.1
28	22.053	22.053	54106	313429	0.24372	RONNEL	6.4
29	24.124	24.124	105649	615384	0.64651	MALATHION	5.9
30	24.711	24.711	58915	350937	0.25479	FENTHION	6.4
31	24.924	24.933	74398	680824	0.47753	CHLORPYRIFOS/ETHYL PARATHION	9.1
32	25.467	0.000	1105	5083	0.00000		3.2
33	25.858	25.867	46087	295370	0.23676	TRICHLORONATE	6.4

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL/L/kg	Peak name	Width
34	27.058	27.049	108634	701174	0.48816	ETHYL PIRIMIPHOS	6.4
35	27.902	27.902	89713	456344	0.67962	MERPHOS	5.3
36	29.182	0.000	6977	23859	0.00000		4.8
37	29.404	0.000	1267	4256	0.00000		4.8
38	29.627	29.627	181324	584632	0.74772	TETRACHLORVINPHOS	3.7
39	30.267	30.267	103301	296926	0.24084	PROTHIOPHOS	3.7
40	30.498	30.498	355866	991359	0.62562	DEF	4.3A
41	31.564	31.556	447995	1198803	1.43350	FENSULFOTHION	3.2A
42	31.822	31.813	232316	587716	0.24628	ETHION	3.2A
43	32.089	32.080	127583	328800	0.25023	SULPROFOS	3.2A
44	32.978	32.969	36656	103099		TRIPHENYLPHOSPHATE	3.7
45	34.400	34.391	228894	780958	1.32077	METHYL AZINPHOS	4.3
46	35.920	35.911	95960	374627	0.65785	COUMAPHOS	4.3

Totals			
Unknowns	0	0	N/A
Quantified	4647274	15620266	13.52270
Grand Total	4647274	15620266	13.52270

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... OPP
Run sequence..... VER0422
Calibration..... OPP0422
Internal standard calibration using area
Calibration last modified on 23-APR-1992 at 08:13

Uncalibrated peaks use user factor (0.0000)

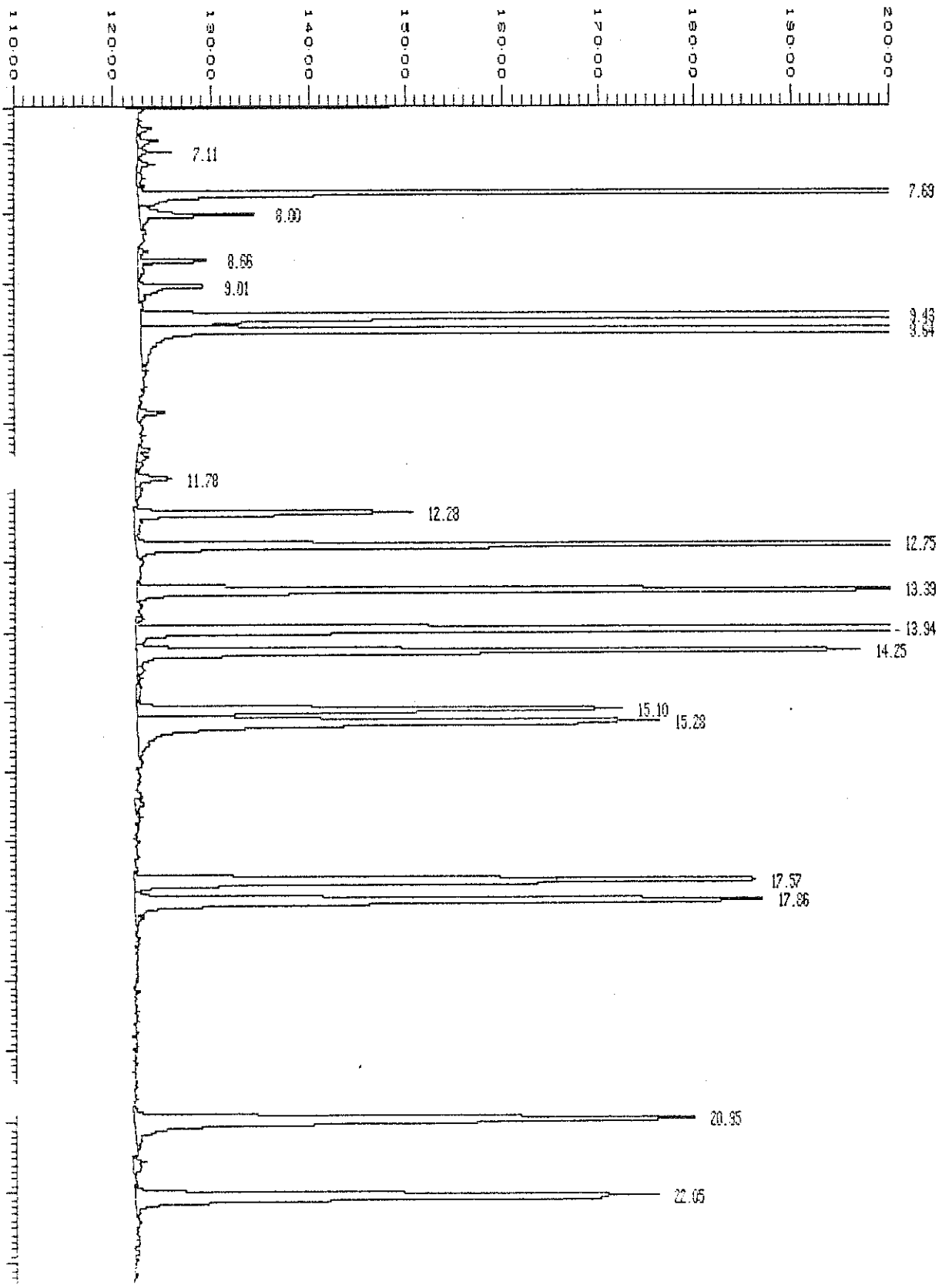
RMAL Chromatography System on DENCR3

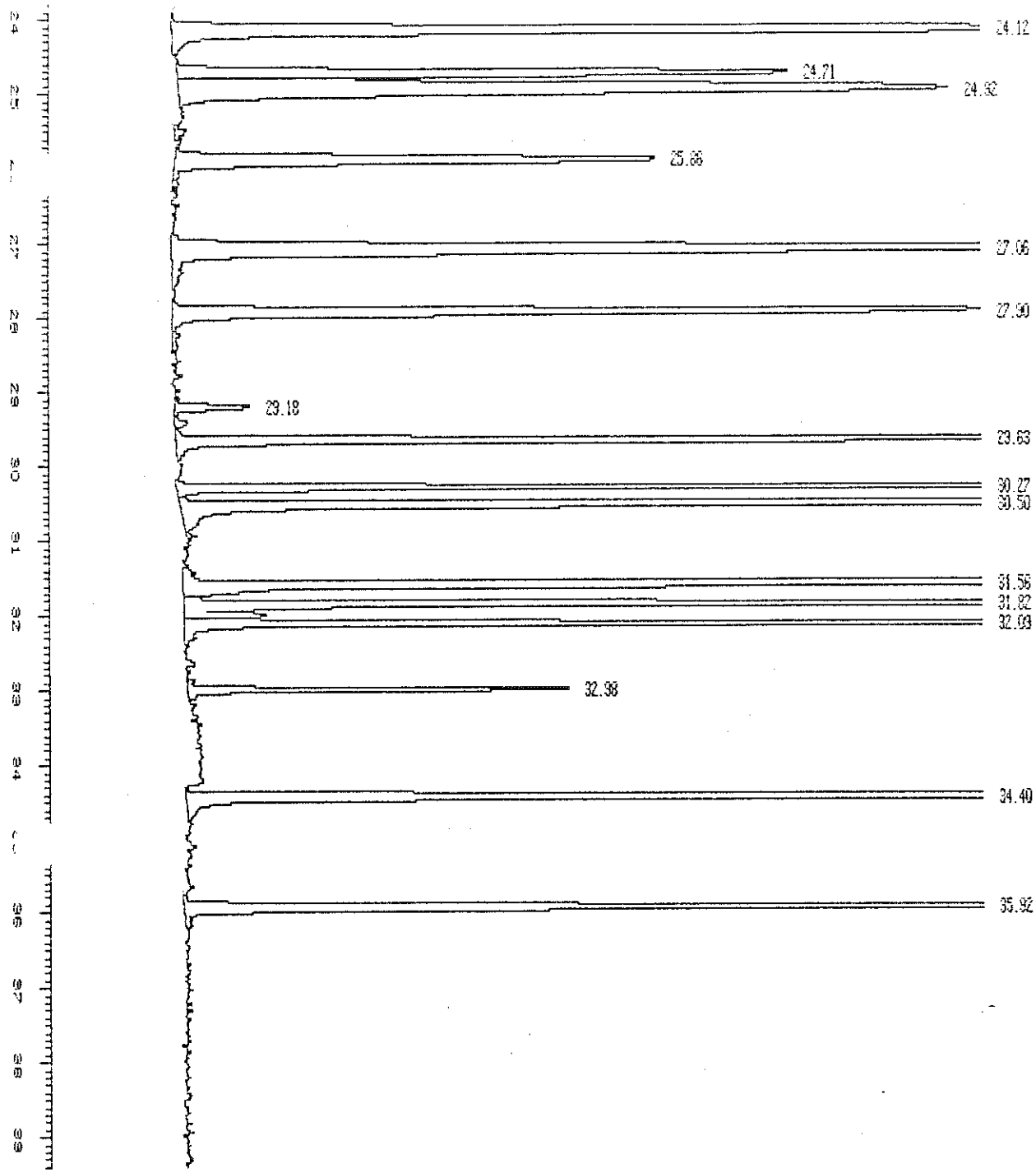
COPP04_3J 52 D22APR92.2.1

8140 CSA 0.5% #5940 T=ST. Amount : 1.000.

Acquired on 22-APR-1992 at 15:36
Reported on 22-APR-1992 at 16:23
Box 1 (of 1)

RE
5-7-92





52
D22APR92,2

AE
5-7-92

Injection Report

Acquired on 22-APR-1992 at 16:24

RMAL Chromatography System on DENCR3

Analyst Name : A.STEYERT
Lims Id : 2 UL INJECT
Comment : D-5890_A, DUAL FPD, RTX-1/RTX-5, 0.25uM, 30 M, 0.25 MM ID
Method Title : OPP 8140
Sample Name : 8140 CSA 0.4% #5940 T=ST
Sample Id :
Sample Type : Standard Amount=1.000000
Bottle No : 3

AG
5-7-92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
1	5.929	0.000	6815	21832	0.00000		5.9
2	6.142	0.000	1693	8445	0.00000		9.6
3	6.480	6.430	550353	888929	0.22169	DICHLORVOS	2.1A
4	6.747	0.000	20459	56408	0.00000		4.3A
5	7.271	0.000	15876	30703	0.00000		2.1A
6	7.484	0.000	6115	26811	0.00000		6.4A
7	7.796	7.796	756669	2230161	0.32423	PHOSDRIN	4.8
8	7.929	7.929	755309	1290148	0.35760	CHLORMEFOS	2.1A
9	8.667	0.000	4994	15923	0.00000		4.3A
10	8.933	0.000	2624	13591	0.00000		3.7
11	9.209	0.000	5220	16358	0.00000		5.3A
12	9.573	0.000	79197	163484	0.00000		3.2A
13	9.804	9.804	270520	597732	0.20701	ETHOPROP	3.2A
14	10.053	10.053	164676	403857	1.47962	NALED	3.2A
15	10.418	10.418	473173	1112376	0.20795	SULFOTEPP	4.3A
16	10.604	10.604	218775	536380	0.20764	PHORATE	3.2A
17	10.764	10.764	139890	496812	0.25050	DIMETHOATE	5.3A
18	10.978	10.978	141437	441079	0.20360	DEMETON (O,S)	3.7
19	11.689	0.000	1670	7974	0.00000		6.4
20	11.902	0.000	1320	4877	0.00000		5.9
21	12.116	0.000	715	3746	0.00000		6.9
C1		12.520	228106	1095815	0.41432	DIAZINON/DISYSTON	
22	12.516	12.520	228106	1095815	0.41432	- C1	5.3
23	14.133	14.133	137598	591286	0.20434	METHYL PARATHION	4.8
24	15.351	15.351	115082	542634	0.20621	RONNEL	5.3
25	16.347	0.000	856	2788	0.00000		3.7
26	16.720	16.720	190857	909973	0.55332	MALATHION	5.3
27	17.040	17.040	110233	554731	0.20717	FENTHION	5.3
28	17.307	17.307	111301	555715	0.21580	ETHYL PARATHION	5.3
29	17.449	17.449	93418	489502	0.19570	CHLORPYRIFOS	5.9
30	17.778	0.000	2218	11526	0.00000		5.9
31	18.569	18.569	87456	461867	0.20259	TRICHLORONATE	5.9
32	19.404	19.404	189926	972649	0.41189	ETHYL PIRIMIPHOS	5.3

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
33	19.751	0.000	1567	5493	0.00000		3.7
34	20.516	20.516	108271	574619	0.53575	MERPHOS	5.9
35	21.813	0.000	6158	34932	0.00000		6.4
36	22.089	22.089	112957	656580	0.59523	TETRACHLORVINPHOS	5.9
37	22.551	0.000	3838	24820	0.00000		6.4
38	23.609	0.000	511	1715	0.00000		6.4
39	23.831	23.831	68165	396472	0.20476	PROTHIOPHOS	6.4
40	24.489	24.489	246496	1418325	0.50455	DEF	6.4
41	25.484	0.000	926	3517	0.00000		5.3
42	26.347	26.347	227246	1760334	1.20095	FENSULFOTHION	7.5
43	27.822	0.000	4223	26582	0.00000		5.9
44	28.062	28.062	178788	817777	0.20430	ETHION	4.8
45	28.578	28.578	115221	481590	0.20426	SULPROFOS	4.8
46	29.467	0.000	594	2270	0.00000		3.7
47	29.680	0.000	1239	8154	0.00000		6.9
48	30.036	30.036	95513	247342		TRIPHENYLPHOSPHATE	3.2A
49	30.702	0.000	829	5090	0.00000		5.9
50	31.413	31.413	694242	1599078	1.18774	METHYL AZINPHOS	3.2A
51	32.996	32.996	318701	715228	0.55920	COUMAPHOS	3.2A
52	34.027	0.000	1742	4170	0.00000		3.7
53	34.373	0.000	874	6788	0.00000		5.3
54	34.969	0.000	405	314	0.00000		3.2

Totals			
Unknowns	0	0	N/A
Quantified	7073057	23347300	11.26792
Grand Total	7073057	23347300	11.26792

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... OPP
Run sequence..... VER0422
Calibration..... OPP0422
Internal standard calibration using area
Calibration last modified on 23-APR-1992 at 08:11

Uncalibrated peaks use user factor (0.0000)

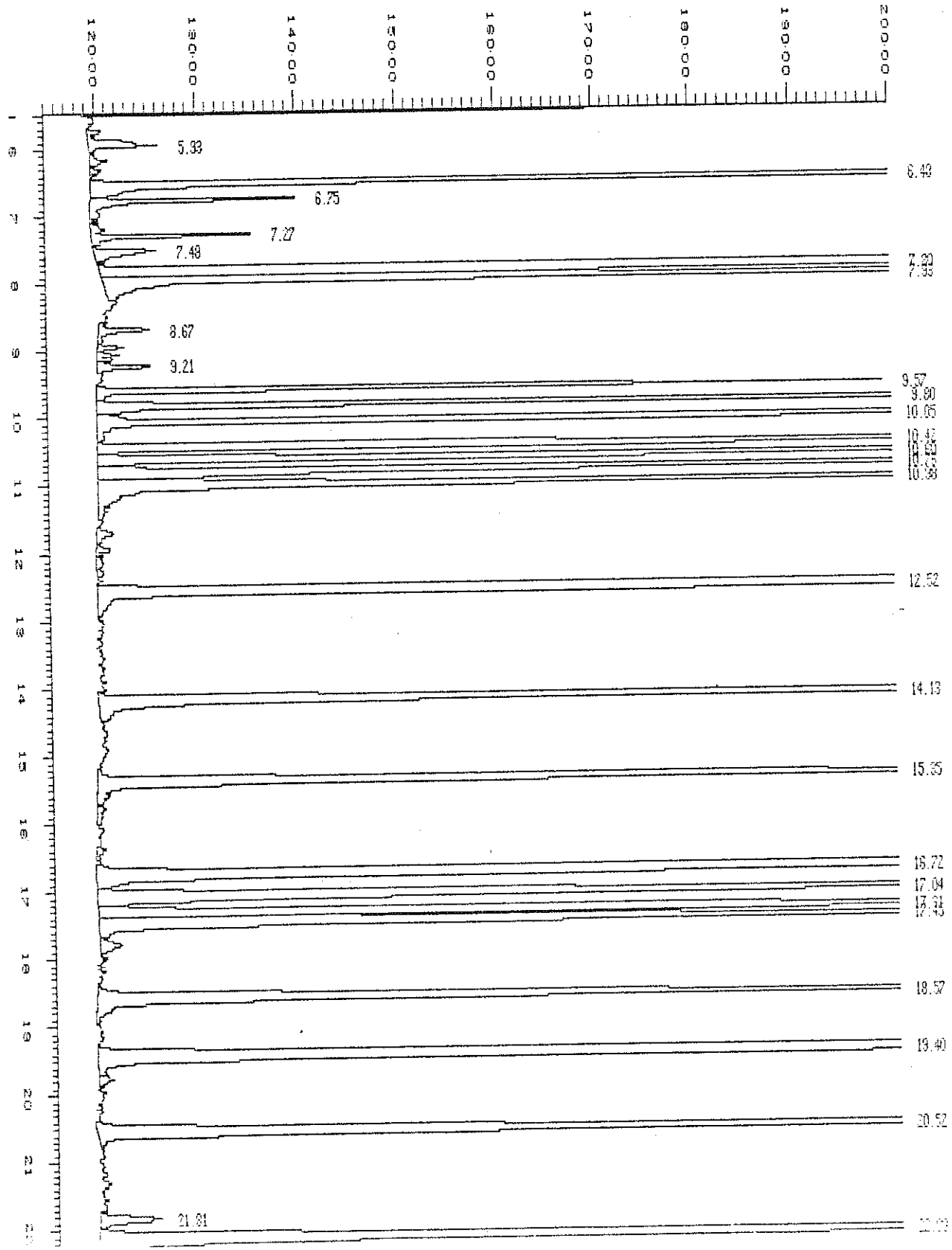
RMAL Chromatography System on DENCR3

COPP04_31 S1 D22APR92,3,1

8140 CSA 0.4% #5940 T=ST. Amount : 1.000.

RE
5-7-92

Acquired on 22-APR-1992 at 16:24
Reported on 22-APR-1992 at 17:11
Box 1 (of 1)



23.93
24.49
26.35
27.82
28.06
28.59
30.04
31.41
33.00

Injection Report

Acquired on 22-APR-1992 at 16:24

RMAL Chromatography System on DENCR3

Analyst Name : A.STEYERT
Lims Id : 2 UL INJECT
Comment : D-5890_A, DUAL FPD, RTX-1/RTX-5, 0.25uM, 30 M, 0.25 MM ID
Method Title : OPP 8140
Sample Name : 8140 CSA 0.4% #5940 T=ST
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 3

RE
5-7-92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL/L/kg	Peak name	Width
1	6.942	0.000	2213	6756	0.00000		4.3
2	7.111	0.000	2475	6345	0.00000		4.3
3	7.271	0.000	927	2343	0.00000		3.2A
4	7.680	7.680	330989	588032	0.22203	DICHLORVOS	3.2A
5	7.991	0.000	9616	31920	0.00000		4.3A
6	8.658	0.000	5059	15447	0.00000		2.1A
7	9.004	0.000	5486	24332	0.00000		4.3
8	9.422	9.422	752590	1833060	0.35483	PHOSDRIN	3.2A
9	9.636	9.636	302674	706852	0.40284	CHLORMEFOS	3.2A
10	10.318	0.000	2019	8891	0.00000		4.3
11	11.369	0.000	1181	4748	0.00000		6.4
12	11.476	0.000	1407	7875	0.00000		4.3
13	11.778	0.000	3034	16459	0.00000		5.3A
14	12.284	0.000	23874	34864	0.00000		4.3
15	12.747	12.747	82346	295068	0.20900	ETHOPROP	4.3
16	13.378	13.378	70319	266966	1.58427	NALED	4.3
17	13.938	13.938	149296	570317	0.21169	SULFOTEPP	4.3
18	14.249	14.249	67424	286930	0.21875	PHORATE	4.3
19	15.102	15.102	43959	199830	0.21778	DEMETON (O,S)	4.8
20	15.289	15.289	47734	292557	0.24138	DIMETHOATE	5.9
21	17.564	17.564	54142	261164	0.20196	DIAZINON	5.3
22	17.849	17.849	54597	278913	0.20221	DISYSTON	5.9
23	20.942	20.942	48316	292155	0.21057	METHYL PARATHION	6.4
24	22.053	22.053	44064	259704	0.20143	RONNEL	5.9
25	24.124	24.124	88279	494812	0.51851	MALATHION	5.9
26	24.711	24.711	49166	287679	0.20833	FENTHION	6.4
27	24.933	24.933	61819	570067	0.39882	CHLORPYRIFOS/ETHYL PARATHION	9.6
28	25.476	0.000	935	8296	0.00000		6.4
29	25.867	25.867	38558	249791	0.19972	TRICHLORONATE	6.4
30	27.049	27.049	88184	567373	0.39400	ETHYL PIRIMIPHOS	6.4
31	27.902	27.902	74540	362962	0.53916	MERPHOS	5.3
32	29.182	0.000	5110	18522	0.00000		4.3
33	29.627	29.627	151687	472133	0.60229	TETRACHLORVINPHOS	4.3A

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL/L/kg	Peak name	Width
34	30.267	30.267	83987	247167	0.19997	PROTHIOPHOS	3.2A
35	30.498	30.498	296714	823496	0.51836	DEF	3.2A
36	31.556	31.556	364401	964894	1.15085	FENSULFOTHION	3.2A
37	31.813	31.813	184327	480783	0.20095	ETHION	4.3A
38	32.080	32.080	107044	263269	0.19984	SULPROFOS	3.2A
39	32.969	32.969	37714	103364		TRIPHENYLPHOSPHATE	3.2A
40	34.391	34.391	190136	662899	1.11824	METHYL AZINPHOS	3.7
41	35.911	35.911	78926	309518	0.54213	COUMAPHOS	4.3
42	37.218	0.000	728	4884	0.00000		20.3

Totals			
Unknowns	0	0	N/A
Quantified	4013496	13233437	11.26996
Grand Total	4013496	13233437	11.26996

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... OPP
Run sequence..... VER0422
Calibration..... OPP0422
Internal standard calibration using area
Calibration last modified on 23-APR-1992 at 08:13

Uncalibrated peaks use user factor (0.0000)

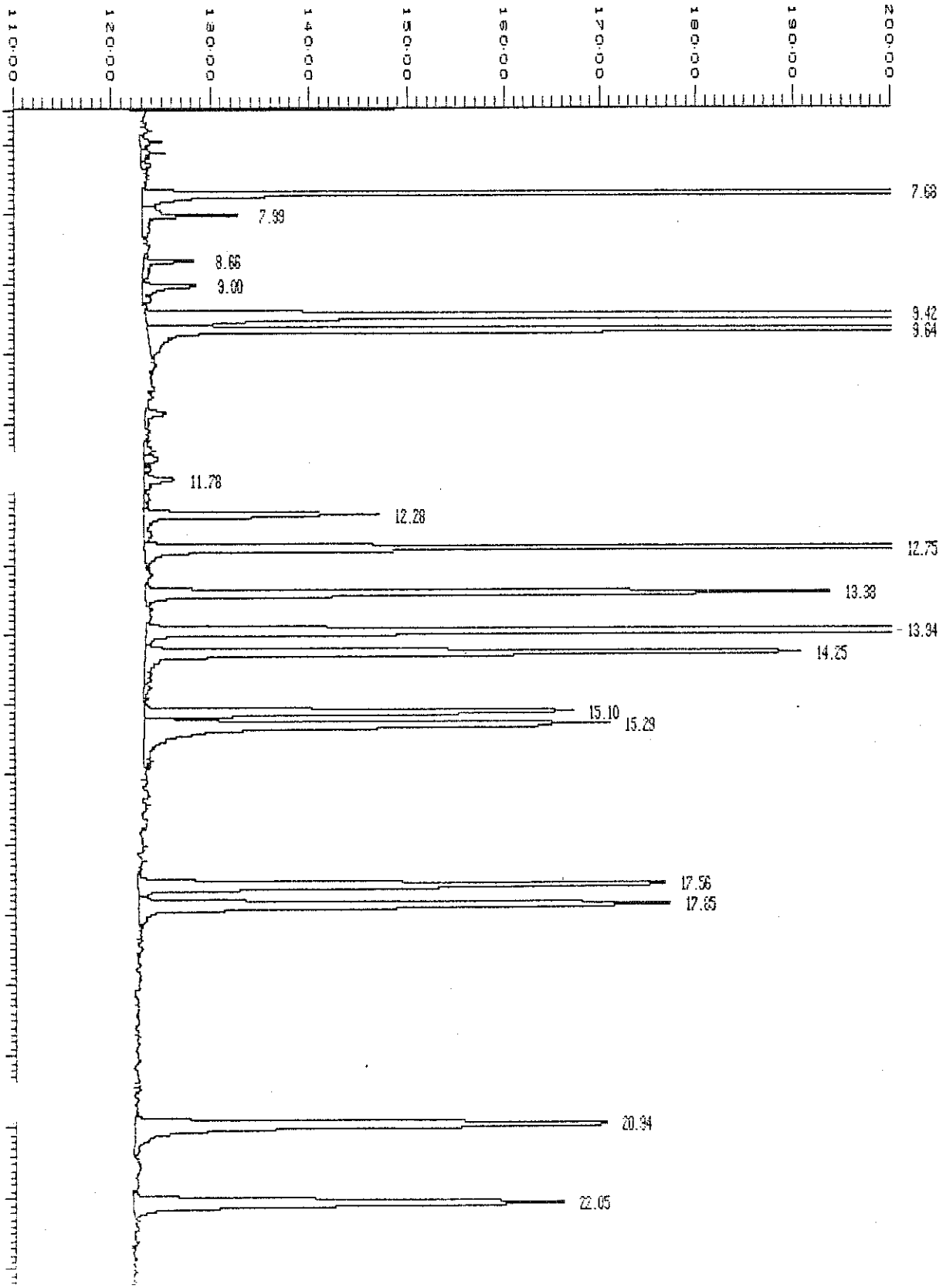
RMAL Chromatography System on DENCR3

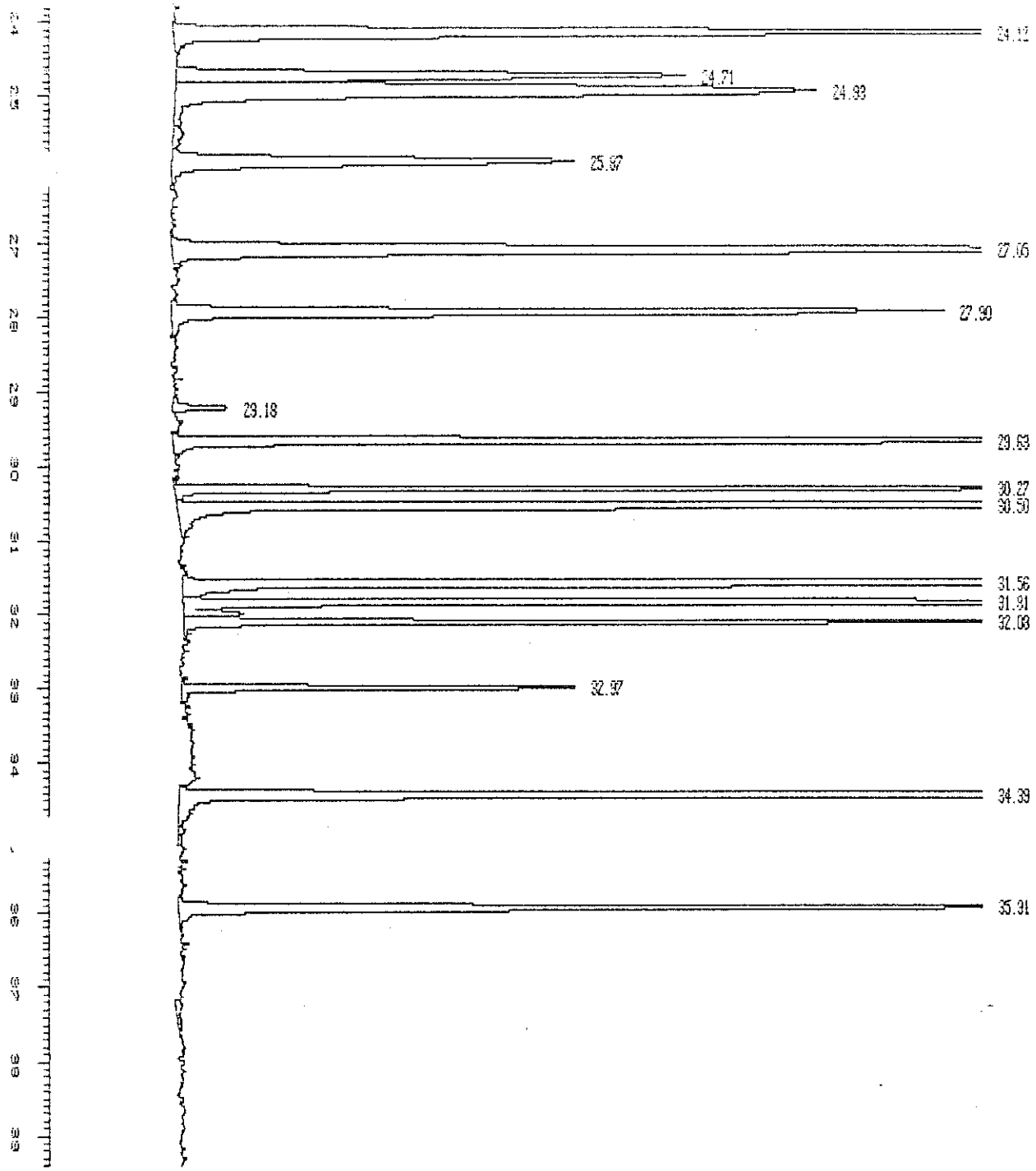
COPP04_3I 52 D22APR92,3,1

AE
5-7-92

3140 CSA 0.4% #5940 T=ST. Amount : 1.000.

Acquired on 22-APR-1992 at 16:24
Reported on 22-APR-1992 at 17:11
Box 1 (of 1)

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32.03
32.97
34.39
35.91

Injection Report

Acquired on 22-APR-1992 at 17:13

RMAL Chromatography System on DENCR3

Analyst Name : A.STEYERT
Lims Id : 2 UL INJECT
Comment : D-5890_A, DUAL FPD, RTX-1/RTX-5, 0.25uM, 30 M, 0.25 MM ID
Method Title : OPP 8140
Sample Name : 8140 CSA 0.2% #5940 T=ST
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 4

RE
5.790

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
1	5.724	0.000	1331	3827	0.00000		4.3
2	5.938	0.000	3223	11595	0.00000		8.5
3	6.169	0.000	1540	7311	0.00000		10.1
4	6.489	6.480	252463	446081	0.10834	DICHLORVOS	3.2A
5	6.756	0.000	8923	29268	0.00000		5.3A
6	7.280	0.000	7629	15343	0.00000		3.2A
7	7.520	0.000	2167	14908	0.00000		7.5
8	7.804	7.796	628948	1328117	0.18805	PHOSDRIN	3.2A
9	7.938	7.929	416631	767599	0.20721	CHLORMEFOS	3.2A
10	8.684	0.000	2243	25836	0.00000		5.3
11	9.244	0.000	2177	10727	0.00000		5.3
12	9.591	0.000	35736	82136	0.00000		2.1A
13	9.822	9.804	126487	297242	0.10025	ETHOPROP	3.2A
14	10.062	10.053	53387	140567	0.50155	NALED	3.2A
15	10.436	10.418	243230	557074	0.10142	SULFOTEPP	3.2A
16	10.613	10.604	106824	267983	0.10103	PHORATE	3.2A
17	10.800	10.764	52910	217635	0.10687	DIMETHOATE	4.8
18	11.004	10.978	62943	212863	0.09569	DEMETON (O,S)	4.3A
C1		12.520	112541	546332	0.20117	DIAZINON/DISYSTON	
19	12.533	12.520	112541	546332	0.20117	- C1	5.3
20	14.151	14.133	64845	303235	0.10206	METHYL PARATHION	4.8
21	14.907	0.000	884	3561	0.00000		3.7
22	15.369	15.351	56160	263671	0.09758	RONNEL	5.3
23	16.738	16.720	90326	437656	0.25918	MALATHION	5.3
24	17.058	17.040	55638	278189	0.10118	FENTHION	5.9
25	17.324	17.307	54647	279013	0.10552	ETHYL PARATHION	5.3
26	17.458	17.449	48295	254638	0.09914	CHLORPYRIFOS	5.3
27	17.760	0.000	1215	4687	0.00000		3.7
28	18.284	0.000	649	2650	0.00000		5.9
29	18.578	18.569	43034	235142	0.10045	TRICHLORONATE	5.9
30	19.422	19.404	95293	493218	0.20341	ETHYL PIRIMIPHOS	5.9
31	20.533	20.516	52580	283954	0.25784	MERPHOS	5.3
32	21.173	0.000	569	373	0.00000		2.1A

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
33	21.322	0.000	3346	16126	0.00000		4.8
34	22.116	22.089	48417	295592	0.26098	TETRACHLORVINPHOS	6.4
35	22.569	0.000	2024	12796	0.00000		6.4
36	23.849	23.831	34583	201736	0.10147	PROTHIOPHOS	6.4
37	24.507	24.489	124061	732912	0.25392	DEF	6.4
38	25.031	0.000	584	1081	0.00000		5.3
39	26.391	26.347	84754	779842	0.51814	FENSULFOTHION	8.5
40	27.858	0.000	2560	19587	0.00000		6.9
41	28.071	28.062	89605	422738	0.10285	ETHION	4.8
42	28.587	28.578	58658	257796	0.10649	SULPROFOS	4.8
43	30.044	30.036	99842	253972		TRIPHENYLPHOSPHATE	3.7
44	31.164	0.000	1209	2488	0.00000		4.8
45	31.431	31.413	300941	739926	0.53524	METHYL AZINPHOS	3.2A
46	33.013	32.996	140900	335583	0.25553	COUMAPHOS	3.2A

Totals			
Unknowns	0	0	N/A
Quantified	3676950	11894605	5.17257
Grand Total	3676950	11894605	5.17257

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... OPP
Run sequence..... VER0422
Calibration..... OPP0422
Internal standard calibration using area
Calibration last modified on 23-APR-1992 at 08:11

Uncalibrated peaks use user factor (0.0000)

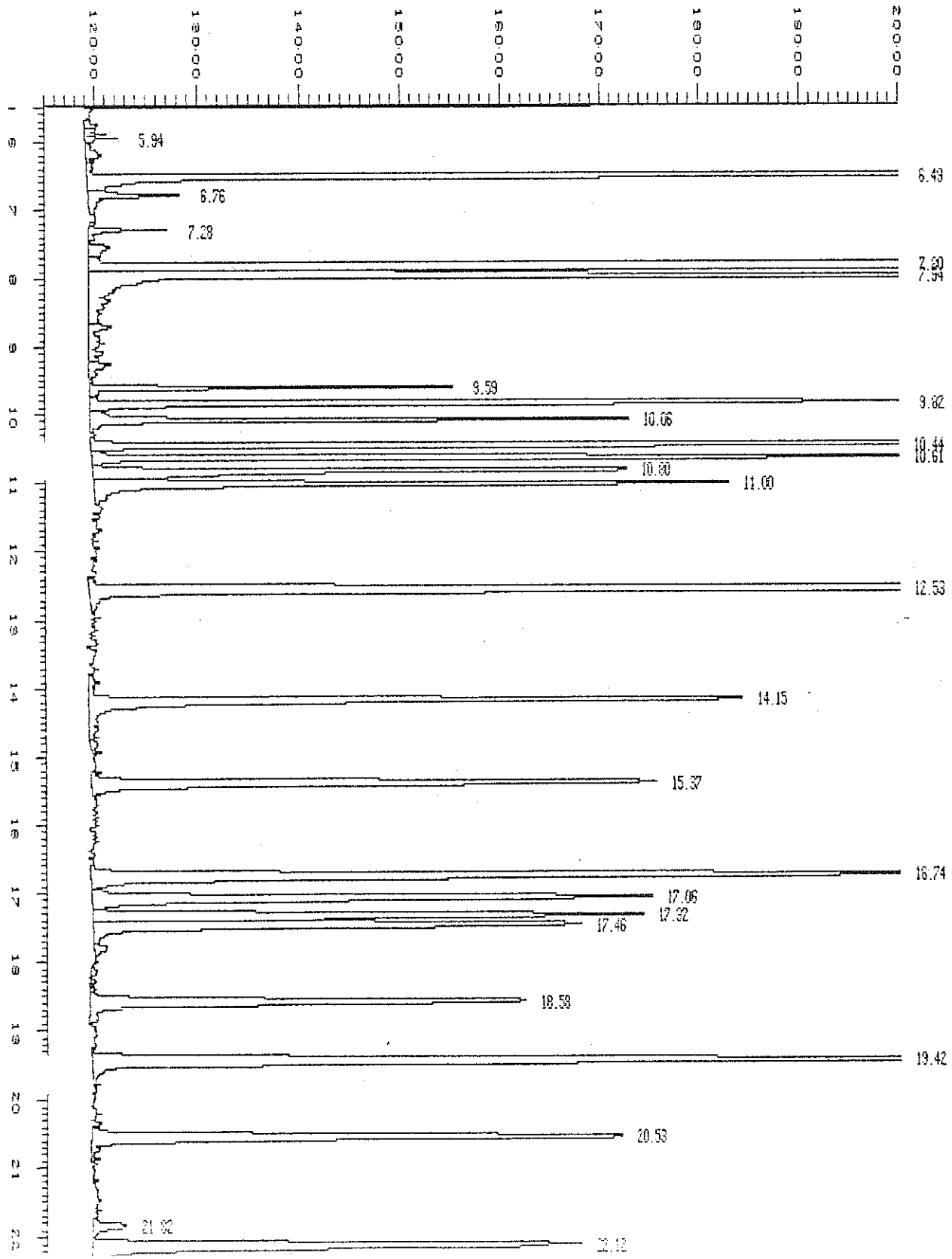
RMAL Chromatography System on DENCR3

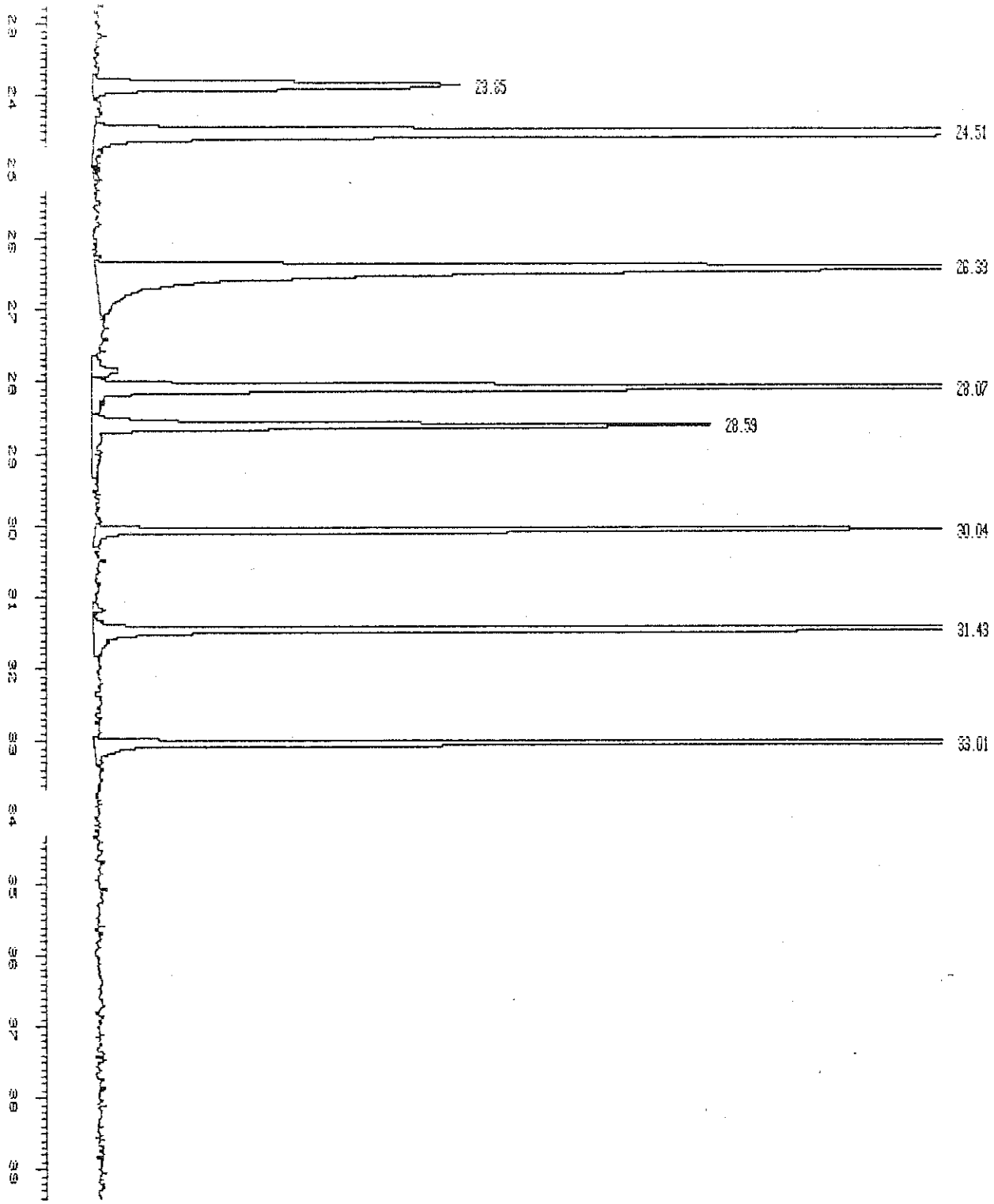
COPP04_3] 51 D22APR92,4,1

8140 CSA 0.2% #5940 T=ST. Amount : 1.000.

Acquired on 22-APR-1992 at 17:13
Reported on 22-APR-1992 at 18:00
Box 1 (of 1)

RE
5-7-92

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Injection Report

Acquired on 22-APR-1992 at 17:13

RMAL Chromatography System on DENCR3

Analyst Name : A.STEYERT
Lims Id : 2 UL INJECT
Comment : D-5890_A, DUAL FPD, RTX-1/RTX-5, 0.25uM, 30 M, 0.25 MM ID
Method Title : OPP 8140
Sample Name : 8140 CSA 0.2% #5940 T=ST
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 4

AE
5-7-92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL/L/kg	Peak name	Width
1	7.129	0.000	1487	6033	0.00000		6.4
2	7.698	7.680	147192	292376	0.10499	DICHLORVOS	3.2A
3	8.018	0.000	4428	13042	0.00000		3.2A
4	8.667	0.000	2818	5833	0.00000		3.2A
5	9.040	0.000	1973	9095	0.00000		5.9
6	9.440	9.422	367634	875898	0.16127	PHOSDRIN	3.2A
7	9.653	9.636	145107	357257	0.19364	CHLORMEFOS	2.1A
8	10.836	0.000	1152	4609	0.00000		8.0
9	11.796	0.000	969	5382	0.00000		8.5
10	12.293	0.000	11241	38399	0.00000		3.7
11	12.764	12.747	39735	147601	0.09943	ETHOPROP	4.3
12	13.404	13.378	22390	88434	0.49911	NALED	4.8
13	13.947	13.938	73827	284153	0.10031	SULFOTEPP	4.8
14	14.258	14.249	31552	132935	0.09639	PHORATE	4.8
15	15.129	15.102	21317	96811	0.10034	DEMETON (O,S)	4.8
16	15.324	15.289	19954	130974	0.10277	DIMETHOATE	5.9
17	17.582	17.564	27977	135870	0.09992	DIAZINON	4.8
18	17.867	17.849	27440	149599	0.10315	DISYSTON	5.3
19	20.542	0.000	678	1922	0.00000		4.3
20	20.978	20.942	23257	148022	0.10146	METHYL PARATHION	6.4
21	21.689	0.000	703	3141	0.00000		6.9
22	22.062	22.053	23047	142369	0.10502	RONNEL	6.4
23	24.142	24.124	42887	252973	0.25211	MALATHION	5.9
24	24.729	24.711	24245	146238	0.10071	FENTHION	6.4
25	24.942	24.933	29760	297562	0.19798	CHLORPYRIFOS/ETHYL PARATHION	10.1
26	25.529	0.000	683	1453	0.00000		3.7
27	25.893	25.867	19334	125499	0.09543	TRICHLORONATE	6.4
28	27.076	27.049	46111	293031	0.19352	ETHYL PIRIMIPHOS	6.9
29	27.920	27.902	36436	180589	0.25512	MERPHOS	5.3
30	29.200	0.000	2871	10589	0.00000		4.3
31	29.644	29.627	66955	219567	0.26638	TETRACHLORVINPHOS	4.3A
32	30.284	30.267	44457	126003	0.09695	PROTHIOPHOS	3.2A
33	30.516	30.498	147047	412197	0.24676	DEF	3.2A

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL/L/kg	Peak name	Width
34	31.582	31.556	153533	447020	0.50707	FENSULFOTHION	3.7
35	31.831	31.813	93816	247473	0.09837	ETHION	4.3A
36	32.098	32.080	53986	138524	0.10000	SULPROFOS	3.2A
37	32.987	32.969	39868	108685		TRIPHENYLPHOSPHATE	3.2A
38	34.204	0.000	910	5199	0.00000		3.7
39	34.418	34.391	94881	326586	0.52394	METHYL AZINPHOS	4.3
40	35.929	35.911	37076	156359	0.26046	COUMAPHOS	4.8
41	38.800	0.000	867	2176	0.00000		3.2A

Totals			
Unknowns	0	0	N/A
Quantified	1931604	6567480	5.06260
Grand Total	1931604	6567480	5.06260

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... OPP
Run sequence..... VER0422
Calibration..... OPP0422
Internal standard calibration using area
Calibration last modified on 23-APR-1992 at 08:13

Uncalibrated peaks use user factor (0.0000)

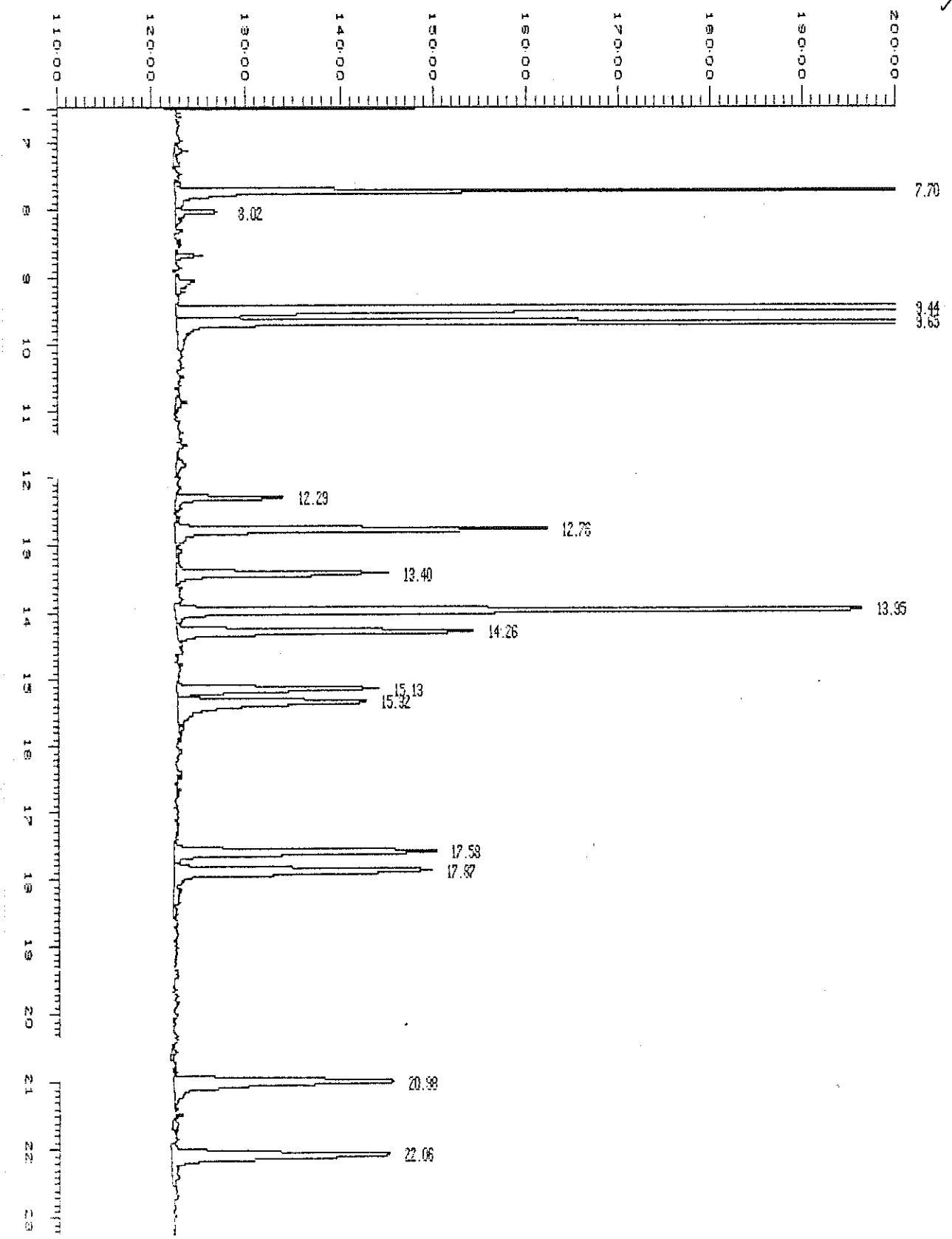
RMAL Chromatography System on DENCR3

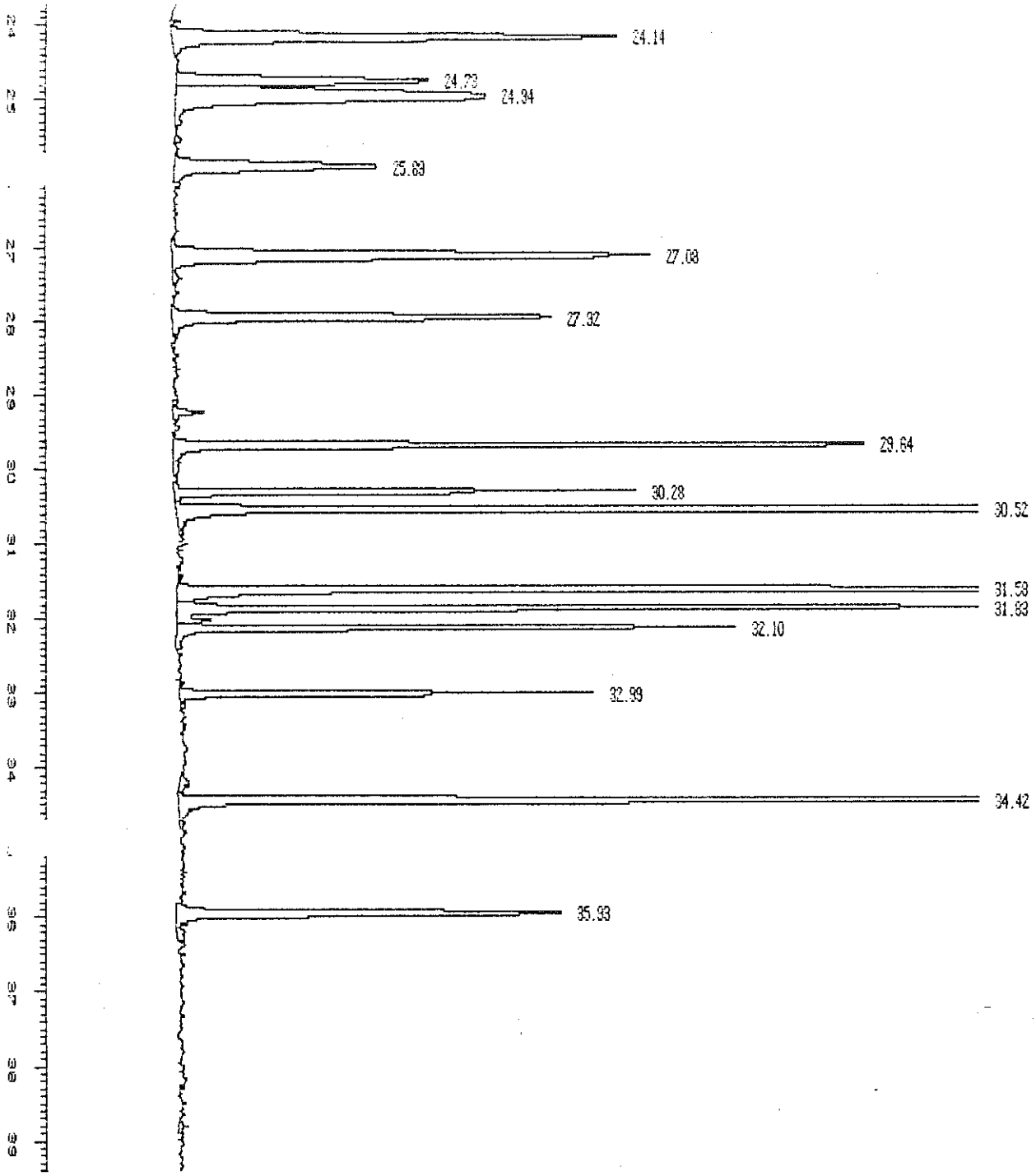
COPP84_33 52 D22APR92,4,1

8146 CSA 0.2% #5940 T=ST. Amount : 1.000.

Acquired on 22-APR-1992 at 17:13
Reported on 22-APR-1992 at 17:59
Box 1 (of 1)

AE
5-7-92

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34.42
35.93

Injection Report

Acquired on 22-APR-1992 at 18:01

RMAL Chromatography System on DENCR3

Analyst Name : A.STEYERT
Lims Id : 2 UL INJECT
Comment : D-5890_A, DUAL FPD, RTX-1/RTX-5, 0.25uM, 30 M, 0.25 MM ID
Method Title : OPP 8140
Sample Name : 8140 CSA 0.1% #5940 T=ST
Sample Id :
Sample Type : Standard Amount=1.000000
Bottle No : 5

AE
5-7-92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
1	5.929	0.000	1772	11979	0.00000		5.3
2	6.480	6.480	85446	183117	0.04507	DICHLORVOS	4.3A
3	6.756	0.000	4219	21792	0.00000		3.2A
4	7.262	0.000	3882	8986	0.00000		6.4
5	7.804	7.796	209283	502469	0.07210	PHOSDRIN	3.2A
6	7.929	7.929	188503	370789	0.10143	CHLORMEFOS	2.1A
7	9.244	0.000	966	3462	0.00000		4.3A
8	9.573	0.000	15456	37717	0.00000		3.2A
9	9.813	9.804	53154	136683	0.04672	ETHOPROP	3.2A
10	10.053	10.053	16922	45520	0.16460	NALED	3.2A
11	10.418	10.418	105160	252545	0.04660	SULFOTEPP	3.7
12	10.604	10.604	47384	122225	0.04670	PHORATE	3.2A
13	10.818	10.764	16249	82318	0.04097	DIMETHOATE	7.5A
14	11.004	10.978	24887	105414	0.04802	DEMETON (O,S)	4.3A
C1		12.520	49947	246918	0.09214	DIAZINON/DISYSTON	
15	12.524	12.520	49947	246918	0.09214	- C1	5.3
16	14.142	14.133	26923	130929	0.04466	METHYL PARATHION	4.8
17	14.880	0.000	813	6001	0.00000		13.9
18	15.351	15.351	24991	122967	0.04612	RONNEL	5.3
19	16.729	16.720	37438	188915	0.11337	MALATHION	5.3
20	17.058	17.040	24175	131535	0.04848	FENTHION	5.9
21	17.307	17.307	24109	122646	0.04701	ETHYL PARATHION	5.9
22	17.440	17.449	22867	128260	0.05061	CHLORPYRIFOS	5.9
23	17.822	0.000	728	3189	0.00000		4.3
24	18.560	18.569	19714	108648	0.04703	TRICHLORONATE	5.9
25	19.404	19.404	43763	229826	0.09605	ETHYL PIRIMIPHOS	5.3
26	20.516	20.516	23750	122872	0.11307	MERPHOS	5.9
27	21.502	0.000	698	2313	0.00000		8.5
28	21.796	0.000	2004	8198	0.00000		6.9
29	22.107	22.089	18480	113105	0.10120	TETRACHLORVINPHOS	6.4
30	22.542	0.000	1092	11068	0.00000		5.9
31	23.093	0.000	922	2834	0.00000		3.7
32	23.831	23.831	16508	98133	0.05002	PROTHIOPHOS	6.4

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
33	24.498	24.489	57158	352875	0.12383	DEF	6.4
34	25.084	0.000	1258	2588	0.00000		4.8
35	26.444	26.347	26183	303530	0.20437	FENSULFOTHION	9.6
36	27.840	0.000	1046	4206	0.00000		2.1
37	28.062	28.062	42545	195248	0.04814	ETHION	5.3
38	28.578	28.578	27846	116234	0.04866	SULPROFOS	4.8
39	30.036	30.036	91875	250613		TRIPHENYLPHOSPHATE	3.2A
40	31.413	31.413	116604	293639	0.21526	METHYL AZINPHOS	3.7
41	32.996	32.996	52910	148679	0.11473	COUMAPHOS	4.3A

Totals			
Unknowns	0	0	N/A
Quantified	1529631	5330987	2.21700
Grand Total	1529631	5330987	2.21700

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... OPP
Run sequence..... VER0422
Calibration..... OPP0422
Internal standard calibration using area
Calibration last modified on 23-APR-1992 at 08:11

Uncalibrated peaks use user factor (0.0000)

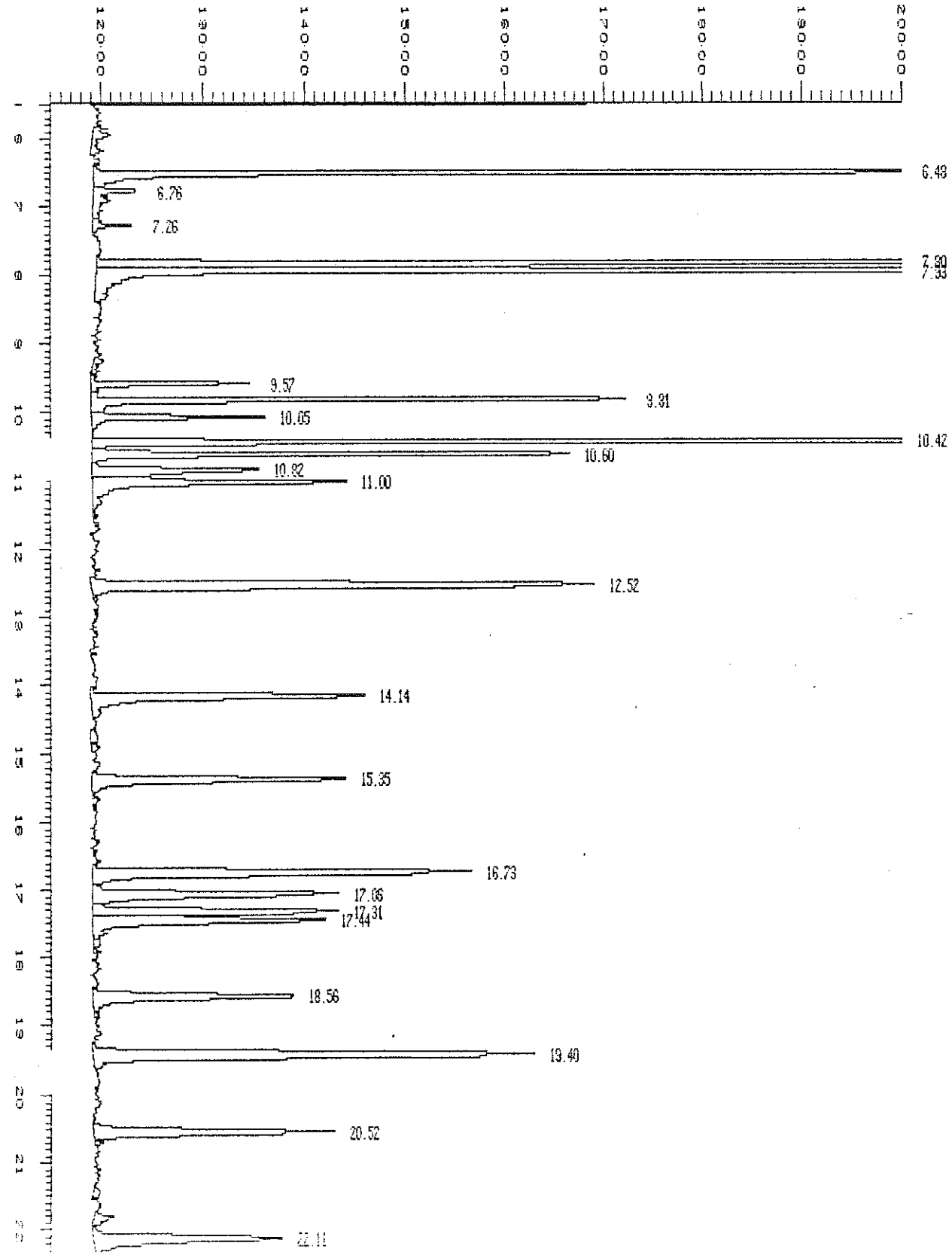
RMAL Chromatography System on DENCR3

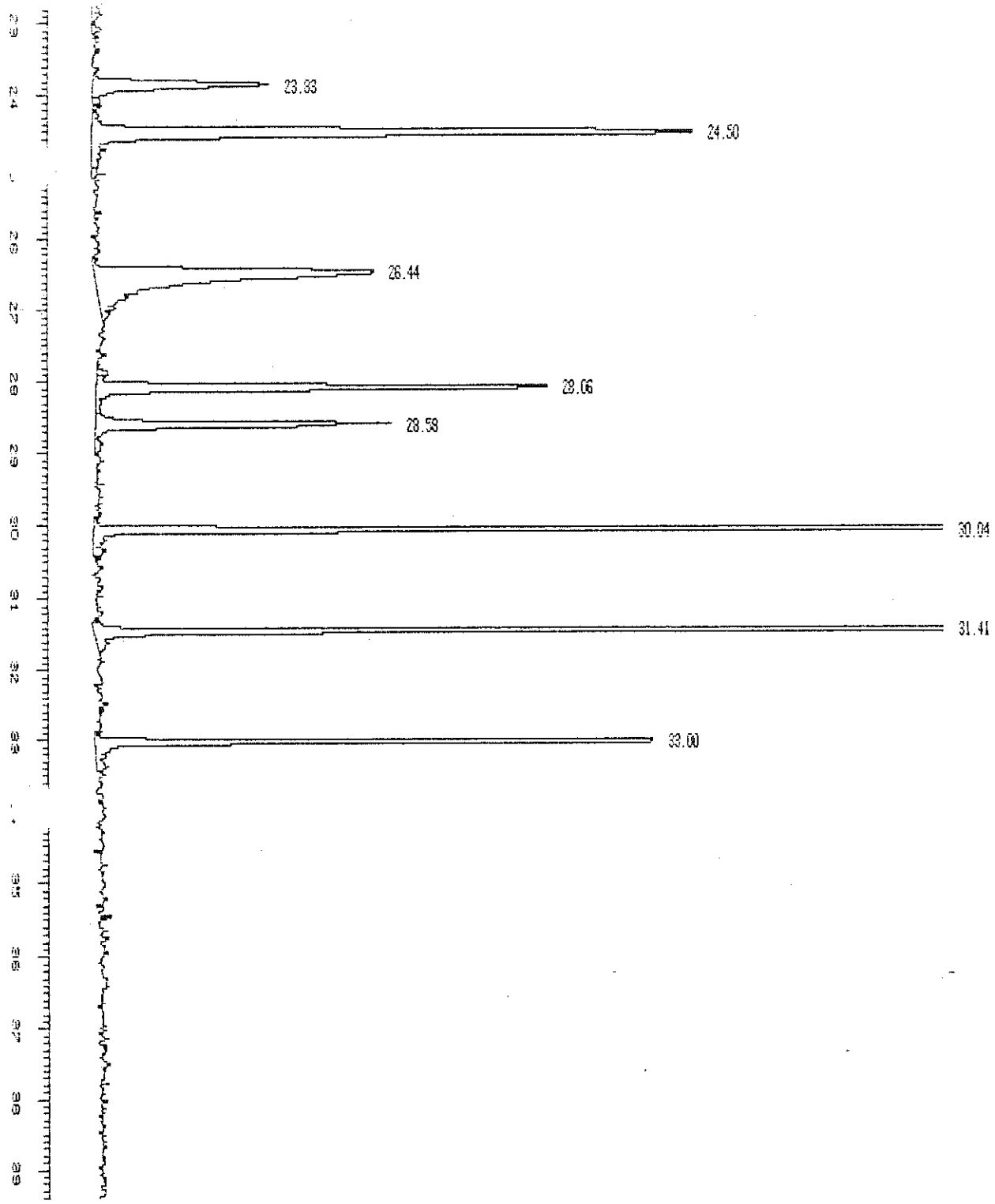
COPP04_3J 51 D22APR92,5,1

3140 CSA 0.1% #5940 T=ST. Amount : 1.000.

Acquired on 22-APR-1992 at 18:01
Reported on 22-APR-1992 at 18:48
Box 1 (of 1)

RE
5-7-92

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Injection Report

Acquired on 22-APR-1992 at 18:01

RMAL Chromatography System on DENCR3

Analyst Name : A.STEYERT
Lims Id : 2 UL INJECT
Comment : D-5890_A, DUAL FPD, RTX-1/RTX-5, 0.25uM, 30 M, 0.25 MM ID
Method Title : OPP 8140
Sample Name : 8140 CSA 0.1% #5940 T=ST
Sample Id :
Sample Type : Standard Amount=1.000000
Bottle No : 5

RE
5-7-92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL/L/kg	Peak name	Width
1	7.716	7.680	54813	117799	0.04465	DICHLORVOS	3.2A
2	8.013	0.000	1890	5014	0.00000		5.3A
3	8.676	0.000	1385	4428	0.00000		2.1A
4	9.120	0.000	1028	7533	0.00000		3.7
5	9.458	9.422	125157	344459	0.06694	PHOSDRIN	3.7
6	9.662	9.636	65434	170356	0.09746	CHLORMEFOS	2.1A
7	12.311	0.000	5330	17880	0.00000		4.3
8	12.782	12.747	17654	70022	0.04979	ETHOPROP	4.3
9	13.413	13.378	6688	27518	0.16392	NALED	4.3
10	13.956	13.938	32662	124742	0.04648	SULFOTEPP	4.8
11	14.276	14.249	14027	63409	0.04853	PHORATE	4.3
12	15.156	15.102	8925	42234	0.04620	DEMETON (O,S)	5.3
13	15.360	15.289	6605	54643	0.04526	DIMETHOATE	7.5
14	17.591	17.564	13079	64020	0.04969	DIAZINON	5.9
15	17.884	17.849	11807	67372	0.04903	DISYSTON	5.3
16	20.996	20.942	10070	72199	0.05223	METHYL PARATHION	6.9
17	22.080	22.053	10416	64476	0.05020	RONNEL	6.4
18	24.160	24.124	17928	113514	0.11940	MALATHION	6.4
19	24.756	24.711	11083	67909	0.04936	FENTHION	6.9
20	24.969	24.933	13333	152756	0.10727	CHLORPYRIFOS/ETHYL PARATHION	10.7
21	25.902	25.867	9297	68080	0.05464	TRICHLORONATE	6.4
22	27.084	27.049	21328	140173	0.09771	ETHYL PIRIMIPHOS	6.4
23	27.929	27.902	15826	77925	0.11619	MERPHOS	5.3
24	29.200	0.000	1701	4815	0.00000		5.3
25	29.662	29.627	25082	83429	0.10683	TETRACHLORVINPHOS	3.7
26	30.293	30.267	21299	59856	0.04861	PROTHIOPHOS	3.2A
27	30.524	30.498	65995	191796	0.12118	DEF	3.2A
28	31.591	31.556	57525	183682	0.21991	FENSULFOTHION	3.7
29	31.840	31.813	43495	115484	0.04845	ETHION	3.2A
30	32.107	32.080	24423	63346	0.04827	SULPROFOS	3.2A
31	32.996	32.969	37835	102974		TRIPHENYLPHOSPHATE	3.7
32	33.689	0.000	723	1965	0.00000		5.9
33	34.427	34.391	37786	139731	0.23660	METHYL AZINPHOS	4.3

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL/L/kg	Peak name	Width
34	35.938	35.911	14870	67340	0.11839	COUMAPHOS	4.8

Totals			
Unknowns	0	0	N/A
Quantified	806499	2952878	2.30319
Grand Total	806499	2952878	2.30319

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... OPP
Run sequence..... VER0422
Calibration..... OPP0422
Internal standard calibration using area
Calibration last modified on 23-APR-1992 at 08:13

Uncalibrated peaks use user factor (0.0000)

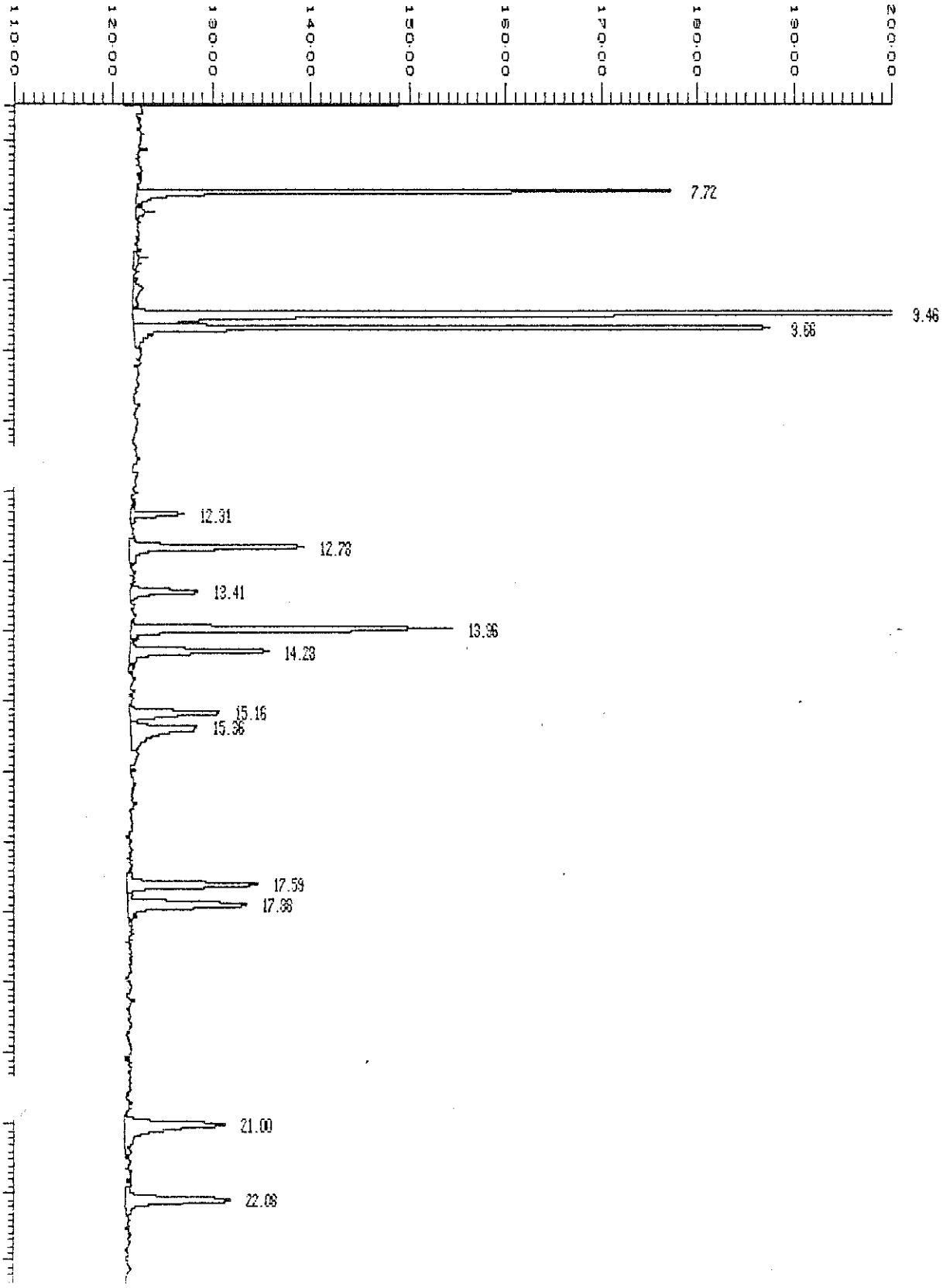
RMAL Chromatography System on DENCR3

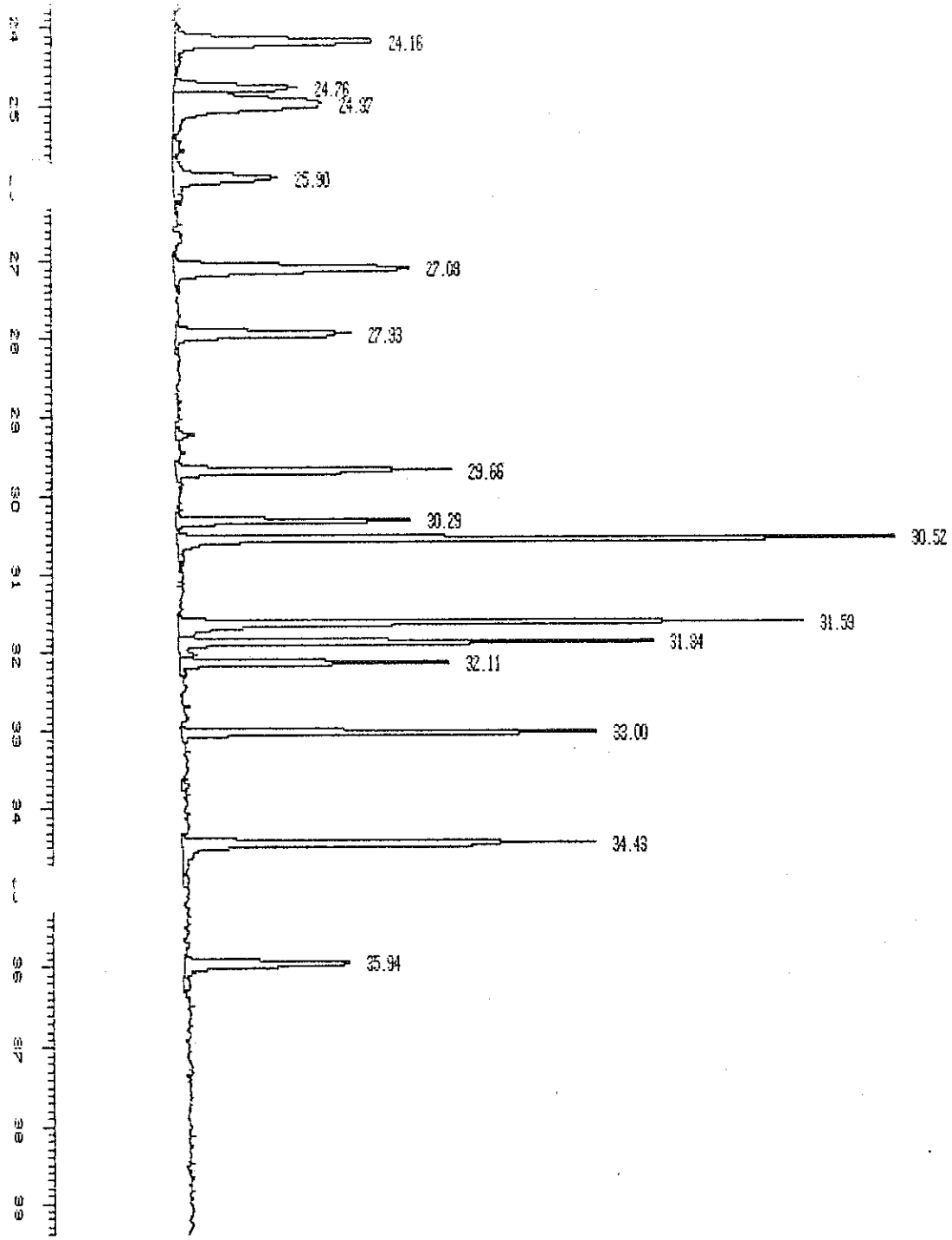
COPP04_3J 52 02APR92,5,1

8140 CSA 0.1% #5940 T=ST. Amount : 1.000.

Acquired on 22-APR-1992 at 18:01
Reported on 22-APR-1992 at 18:47
Box 1 (of 1)

RE 5-7-92

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24.76
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25.90
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30.29
30.52
31.59
31.94
32.11
33.00
34.43
35.94

Injection Report

Acquired on 22-APR-1992 at 18:49

RMAL Chromatography System on DENCR3

Analyst Name : A.STEYERT
Lims Id : 2 UL INJECT
Comment : D-5890_A, DUAL FPD, RTX-1/RTX-5, 0.25uM, 30 M, 0.25 MM ID
Method Title : OPP 8140
Sample Name : 8140 CSA 0.05% #5940 T=ST
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 6

RE 5-7-92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
1	5.938	0.000	1211	9186	0.00000		4.3
2	6.507	6.480	32127	81401	0.02082	DICHLORVOS	4.3A
3	6.782	0.000	1976	6786	0.00000		4.3A
4	7.280	0.000	1651	4639	0.00000		2.1A
5	7.840	7.796	59929	185459	0.02765	PHOSDRIN	4.3
6	7.938	7.929	101388	221234	0.06289	CHLORMEFOS	3.2A
7	9.591	0.000	7558	20728	0.00000		3.2A
8	9.831	9.804	24761	73637	0.02615	ETHOPROP	4.3A
9	10.071	10.053	4008	12622	0.04743	NALED	3.7
10	10.436	10.418	54307	134937	0.02587	SULFOTEPP	3.2A
11	10.622	10.604	23952	65294	0.02592	PHORATE	3.2A
12	10.889	10.764	5709	32275	0.01669	DIMETHOATE	6.4
13	11.040	10.978	10744	59164	0.02801	DEMETON (O,S)	4.8
C1		12.520	24567	137139	0.05318	DIAZINON/DISYSTON	
14	12.533	12.520	24567	137139	0.05318	- C1	5.9
15	14.169	14.133	12215	77295	0.02740	METHYL PARATHION	5.3
16	15.378	15.351	12237	69597	0.02712	RONNEL	5.9
17	16.747	16.720	17508	87821	0.05477	MALATHION	5.3
18	17.067	17.040	12066	63795	0.02443	FENTHION	5.9
19	17.342	17.307	11165	55454	0.02209	ETHYL PARATHION	5.9
20	17.476	17.449	10931	65215	0.02674	CHLORPYRIFOS	6.4
21	17.956	0.000	798	3137	0.00000		5.3
22	18.587	18.569	10284	58852	0.02647	TRICHLORONATE	5.9
23	19.413	19.404	22326	114634	0.04978	ETHYL PIRIMIPHOS	5.3
24	20.542	20.516	10961	60962	0.05829	MERPHOS	5.9
25	21.164	0.000	820	1495	0.00000		2.1
26	21.484	0.000	1069	4086	0.00000		4.8
27	21.831	0.000	1638	9229	0.00000		8.5
28	22.133	22.089	7537	52219	0.04855	TETRACHLORVINPHOS	6.9
29	22.578	0.000	1042	4892	0.00000		6.9
30	23.849	23.831	7428	44290	0.02346	PROTHIOPHOS	6.4
31	24.329	0.000	680	2182	0.00000		2.1
32	24.507	24.489	28313	172455	0.06292	DEF	6.4

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
33	26.551	26.347	9898	135234	0.09462	FENSULFOTHION	12.8
34	27.307	0.000	1163	10103	0.00000		4.8
35	28.030	28.062	21415	97897	0.02508	ETHION	5.3
36	28.596	28.578	13746	56107	0.02441	SULPROFOS	4.8
37	30.044	30.036	86059	241177		TRIPHENYLPHOSPHATE	4.3A
38	30.578	0.000	844	2021	0.00000		3.7
39	31.431	31.413	45193	124127	0.09455	METHYL AZINPHOS	4.3A
40	33.022	32.996	22360	65058	0.05217	COUMAPHOS	3.2A

Totals			
Unknowns	0	0	N/A
Quantified	723666	2723838	1.07745
Grand Total	723666	2723838	1.07745

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... OPP
Run sequence..... VER0422
Calibration..... OPP0422
Internal standard calibration using area
Calibration last modified on 23-APR-1992 at 08:11

Uncalibrated peaks use user factor (0.0000)

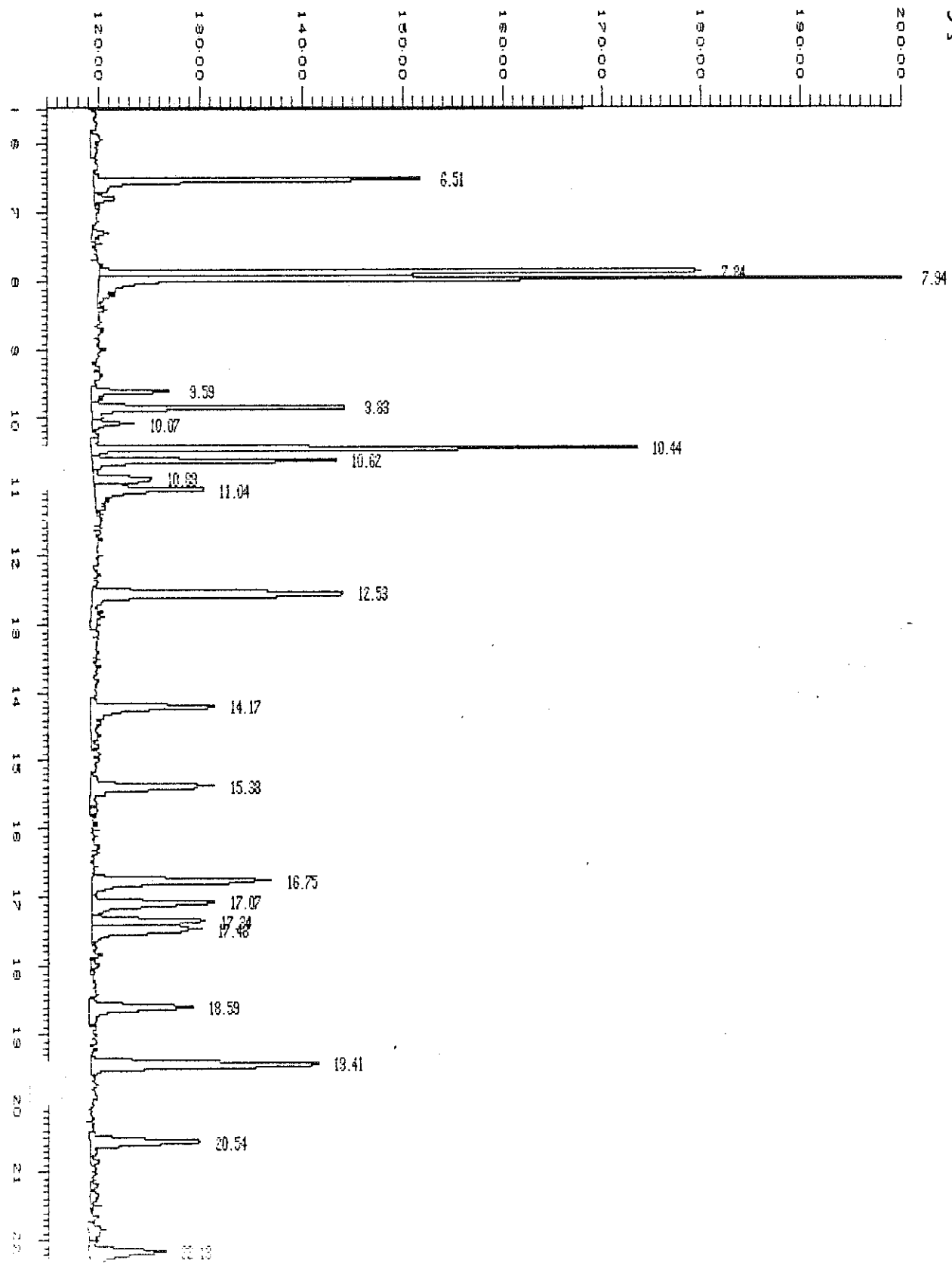
RMAL Chromatography System on DENCR3

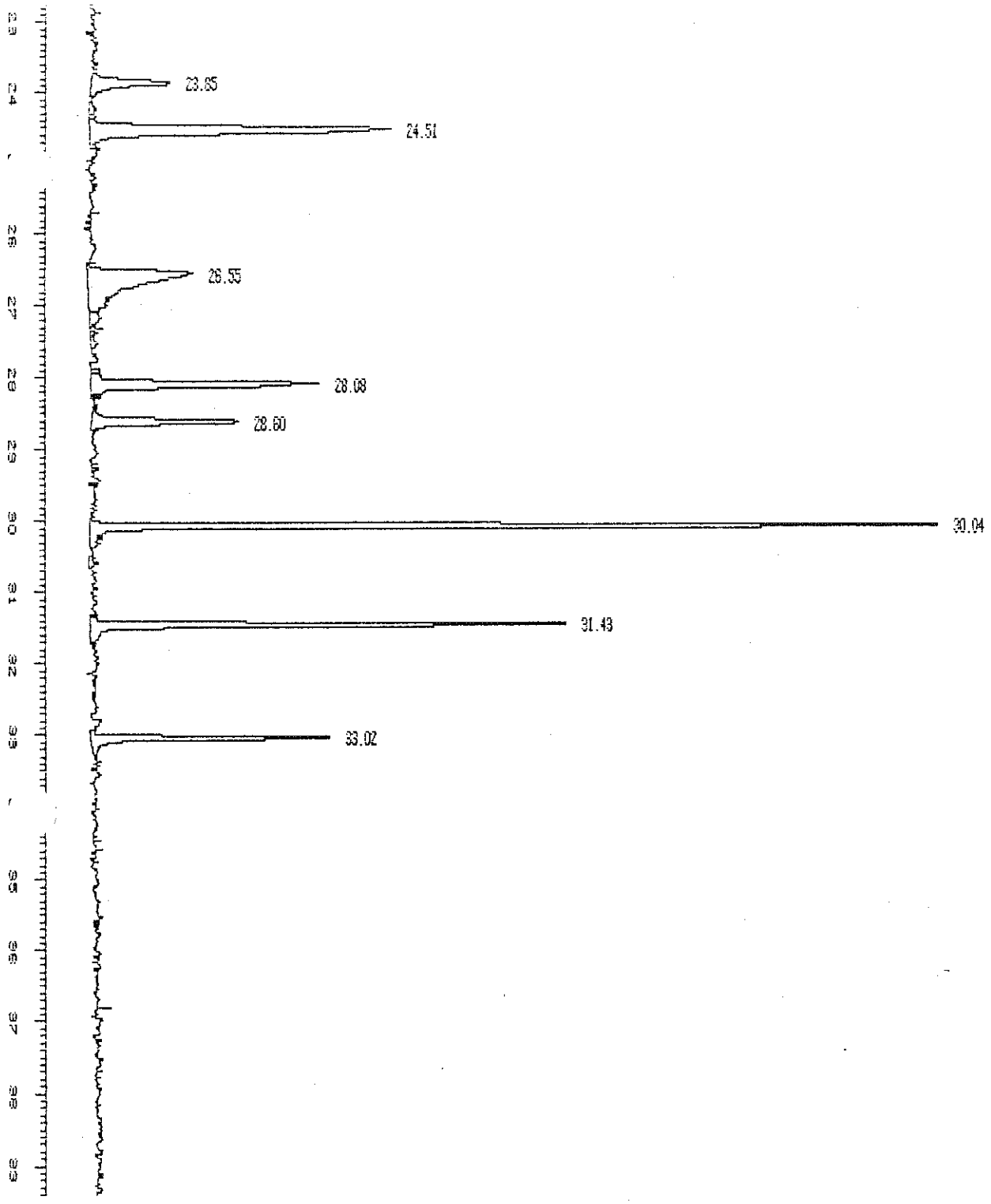
COPP04_31 51 D22APR92,6,1

814w CSA 0.05% #5940 T=ST. Amount : 1.000.

Acquired on 22-APR-1992 at 18:49
Reported on 22-APR-1992 at 19:37
Box 1 (of 1)

RE
5-7-92

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30.04
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Injection Report

Acquired on 22-APR-1992 at 18:49

RMAL Chromatography System on DENCR3

Analyst Name : A.STEYERT
Lims Id : 2 UL INJECT
Comment : D-5890_A, DUAL FPD, RTX-1/RTX-5, 0.25uM, 30 M, 0.25 MM ID
Method Title : OPP 8140
Sample Name : 8140 CSA 0.05% #5940 T=ST
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 6

RE 5-7-92

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	Area uVs	ug/mL/L/kg	Peak name	Width
1	7.707	7.680	22619	57410	0.02263	DICHLORVOS	3.2A
2	9.093	0.000	694	3793	0.00000		5.3
3	9.458	9.422	43188	150260	0.03037	PHOSDRIN	4.3
4	9.644	9.636	35838	92300	0.05493	CHLORMEFOS	3.2A
5	11.804	0.000	648	4738	0.00000		8.0
6	12.302	0.000	2415	8763	0.00000		4.3A
7	12.773	12.747	8493	34489	0.02551	ETHOPROP	4.8
8	13.422	13.378	1837	5163	0.03199	NALED	1.6
9	13.947	13.938	17536	67258	0.02607	SULFOTEPP	4.3
10	14.267	14.249	7138	31631	0.02518	PHORATE	4.3
11	15.156	15.102	3968	21890	0.02491	DEMETON (O,S)	5.3
12	15.387	15.289	3131	23500	0.02025	DIMETHOATE	7.5
13	17.582	17.564	6793	32182	0.02599	DIAZINON	5.3
14	17.867	17.849	6100	33213	0.02514	DISYSTON	5.3
15	21.004	20.942	4494	28974	0.02130	METHYL PARATHION	6.9
16	22.071	22.053	4642	29753	0.02410	RONNEL	7.5
17	24.142	24.124	8801	55116	0.06031	MALATHION	5.9
18	24.729	24.711	5039	31238	0.02362	FENTHION	7.5
19	24.969	24.933	6480	67436	0.04926	CHLORPYRIFOS/ETHYL PARATHION	12.3
20	25.884	25.867	4928	30165	0.02518	TRICHLORONATE	7.5
21	27.067	27.049	11385	75438	0.05470	ETHYL PIRIMIPHOS	6.9
22	27.911	27.902	7052	35632	0.05527	MERPHOS	4.8
23	29.182	0.000	943	3738	0.00000		3.7
24	29.653	29.627	9337	31854	0.04243	TETRACHLORVINPHOS	3.7
25	30.276	30.267	10158	32406	0.02738	PROTHIOPHOS	4.3A
26	30.507	30.498	32604	95642	0.06286	DEF	4.3A
27	31.591	31.556	24457	81156	0.10107	FENSULFOTHION	4.3A
28	31.831	31.813	20635	60571	0.02643	ETHION	3.7
29	32.089	32.080	11502	32629	0.02586	SULPROFOS	4.3A
30	32.978	32.969	36925	98992		TRIPHENYLPHOSPHATE	3.2A
31	34.409	34.391	15634	58963	0.10386	METHYL AZINPHOS	4.3
32	35.929	35.911	7005	29875	0.05464	COUMAPHOS	4.8

Totals			
Unknowns	0	0	N/A
Quantified	382470	1446166	1.07174
Grand Total	382470	1446166	1.07174

MISSING PEAKS

No missing peaks.

ANALYSIS SUMMARY

Method..... OPP
Run sequence..... VER0422
Calibration..... OPP0422
Internal standard calibration using area
Calibration last modified on 23-APR-1992 at 08:13

Uncalibrated peaks use user factor (0.0000)

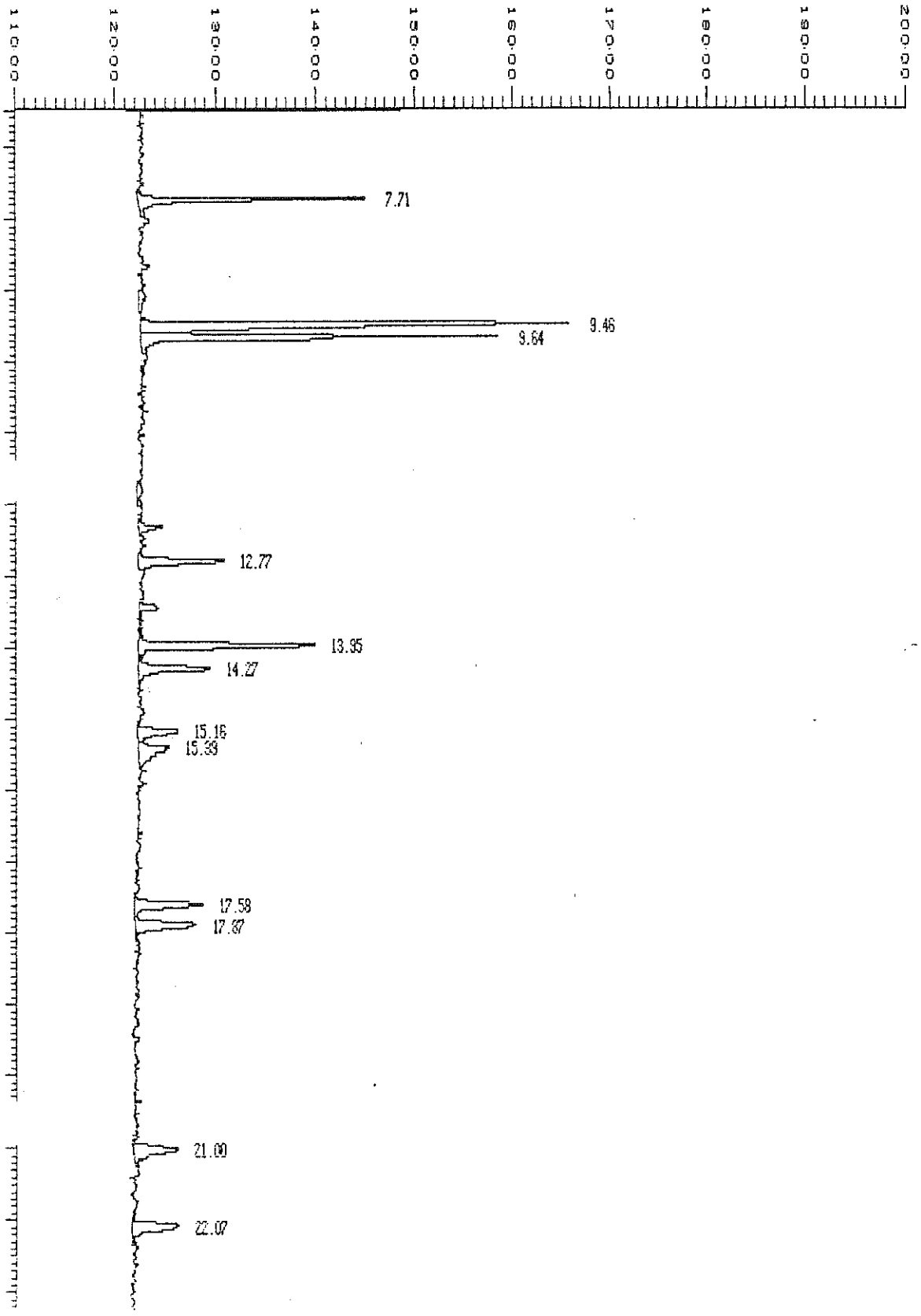
RMAL Chromatography System on DENCR3

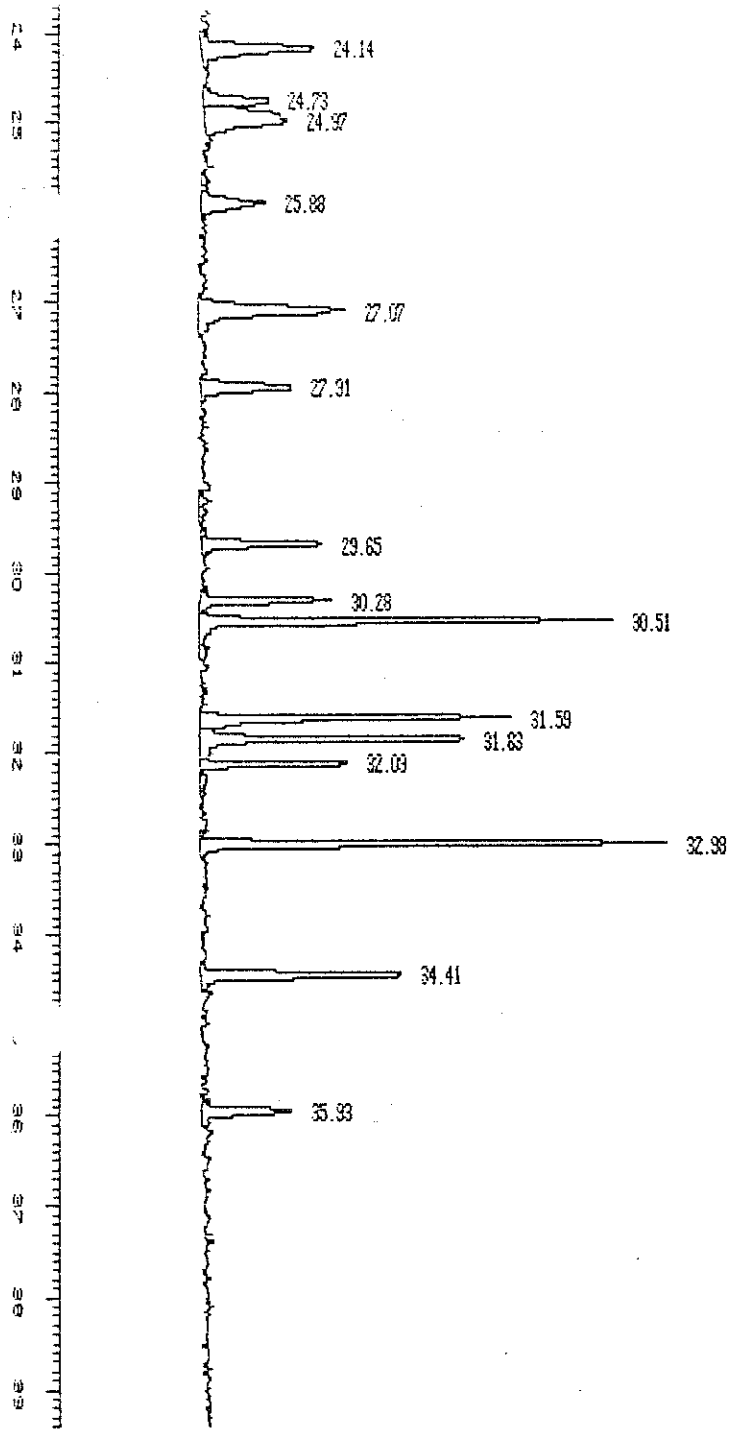
COPP04_3] 52 D22APR92,6,1

8140 CSA 0.05% #5940 T=ST. Amount : 1.000.

Acquired on 22-APR-1992 at 18:49
Reported on 22-APR-1992 at 19:36
Box 1 (of 1)

AE 5-7-92

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25.88
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29.65
30.28
30.51
31.59
31.83
32.09
32.98
34.41
35.33

APPENDIX B-10C - SOPs FOR ORGANOPHOSPHORUS PESTICIDE STANDARD PREPARATION

These SOPs are currently undergoing revision by Enseco. They will be provided when they become available.

This addendum is specific to the Chemical Waste Management, Vickery, Ground Water Monitoring Plan QAPjP. Procedural changes or clarification statements presented herein are not applicable to Enseco SOPs for other projects, and are made here in response to EPA letters dated March 17, 1992 and September 15, 1992 to CWM containing comments (with additional attachments) regarding the CWM-Vickery GWMP QAPjP.

Section 2.1 Enseco-RMAL performs GC analyses using two dissimilar columns in order to accurately identify and quantitate organic compounds. Both columns are individually calibrated and must conform to constraints of ±20% difference for all compounds. When a compound is detected on both columns, the quantitative result is generally chosen from the lower of the two determinations. The higher value can reflect contamination from co-eluters, so the result is generally biased high. Based on this convention, Enseco-RMAL cannot designate one column as the primary column. Since quantitative data is taken from both columns throughout an analysis, both columns are, in a sense, equal to each other during the analysis. Only in extenuating circumstances will deviation from this procedure be this procedure be allowed. In those instances, the scientist will use his/her judgement to make accurate determinations.

Section 2.2: Please refer to QAPjP Section IX for the list of project target compounds and QAPjP Appendix C-1 for the project specific quantitation limits.

Section 7.: Please refer to Section 9.1 of this SOP for additional data on instrument calibration standards and methods. QAPjP Appendix B-11A contains an example of a five point instrument calibration. OCP/PCB target compound linear ranges are presented in a table at the front of QAPjP Appendix B-11A.

Section 8.2.4 and 8.2.5: Please see comment for Section 10.1, below.

Sample Cleanups: GPC and /or Acid cleanups will not be performed for this project since only aqueous samples will be analyzed.

Section 10.1: Matrix spike and matrix spike duplicate analysis are performed at a frequency of one per every group of 20 investigative samples. The compounds and concentrations used are the same as shown for duplicate control samples on pages 27 and 28 of the SOP.

Relative Retention Time:

Parameter retention time is variable with instrument condition and may vary slightly from calibration to calibration. Relative parameter retention times are presented in a table at the front of QAPjP Appendix B-11, immediately following this addendum page. Retention times recorded for an actual calibration are found in QAPjP Appendix B-11A.

Standard Preparation:

Standard preparation SOPs are located in QAPjP Appendix B-11B.

Injection Report

Acquired on 5-OCT-1992 at 15:16

RMAL Chromatography System on DENCR3

Analyst Name : WSULLIVAN
Lims Id : 1uL injected
Comment : T-5880,DUAL ECD Rtx-35,0.53,1.00um, Rtx-5, 0.53,1.5um
Method Title :
Sample Name : Individual Mix AB 0.10UG/ML T=ST
Sample Id : VER #6266
Sample Type : Standard Amount=1.00000
Bottle No : 4

WDS
10/6/92

PEAK INFORMATION

RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
10.169	10.169	6237765	20535902	0.10409	Tetrachloro-m-xylene (TCX)	3.2
11.680	11.676	7906152	29062398	0.10223	alpha-BHC	3.7
12.720	12.720	6609981	26670070	0.10055	gamma-BHC	4.0
12.902	12.902	3188931	13316265	0.10322	beta-BHC	4.0
	14.108	6922389	49745108	0.19863	Heptachlor/delta-BHC	
14.013	14.108	6919711	49737448	0.19860	- C1	7.5
14.227	14.108	2678	7662	0.00102	- C1	3.5
15.084	15.080	4572271	23426022	0.10036	Aldrin	5.1
17.080	17.076	4017109	22628496	0.09732	Heptachlor Epoxide	5.3
18.551	18.547	3515363	20660786	0.09990	Endosulfan I	5.9
19.431	19.427	3631645	19743788	0.10587	4,4'-DDE	5.3
19.804	19.804	3426686	20189360	0.10167	Dieldrin	5.6
21.289	21.289	2785363	16995780	0.10029	Endrin	5.9
21.876	21.876	2674163	15203524	0.10426	4,4'-DDD	5.6
22.120	22.120	2915200	18397630	0.10001	Endosulfan II	6.1
23.333	23.333	2470056	14642960	0.10208	4,4'-DDT	5.9
23.511	23.511	2175128	14220047	0.09818	Endrin Aldehyde	6.1
24.293	24.293	2583194	16799824	0.09747	Endosulfan Sulfate	6.1
26.302	26.298	2221667	13420259	0.09976	Dibutylchlorendate (DBC)	5.9
27.076	27.076	1526864	8229522	0.10103	Methoxychlor	5.1
27.418	27.413	3029922	16208805	0.10492	Endrin Ketone	5.3
33.329	33.329	734935	5986909	0.02612	Octachloronapthalene	8.0
33.631	33.627	1936592	17340786	0.09616	Decachlorobiphenyl (DCB)	8.5

Totals			
Unknowns	1876418	9902333	N/A
Quantified	75081376	403424288	2.14413
Grand Total	76957792	413326624	2.14413

MISSING PEAKS

No missing peaks.

PEAK GROUP INFORMATION

No peak group information.

ANALYSIS SUMMARY

Method..... TOCP
Run sequence..... T1005
Calibration..... T1005
External standard calibration using area
Calibration last modified on 6-OCT-1992 at 08:30

Uncalibrated peaks use user factor (0.0000)

Injection Report

Acquired on 5-OCT-1992 at 15:16

RMAL Chromatography System on DENCR3

Analyst Name : WSULLIVAN
Lims Id : 1uL injected
Comment : T-5880,DUAL ECD Rtx-35,0.53,1.00um, Rtx-5, 0.53,1.5um
Method Title :
Sample Name : Individual Mix AB 0.10UG/ML T=ST
Sample Id : VER #6266
Sample Type : Standard Amount=1.00000
Bottle No : 4

WDS
10/6/92

PEAK INFORMATION

RT mins	RT Exp	Hght uV	Area uVs	ug/mL	Peak name	Width
9.551	9.538	4749407	14345040	0.09630	Tetrachloro-m-xylene (TCX)	2.7
10.258	10.244	7293855	22617136	0.10027	alpha-BHC	2.9
10.742	10.733	3063950	10768210	0.09415	beta-BHC	3.5
10.902	10.893	6374363	21703778	0.09927	gamma-BHC	3.2
11.387	11.373	5884539	20701982	0.10318	delta-BHC	3.7
12.627	12.613	5024434	21726198	0.09669	Heptachlor	4.3
13.618	13.604	4552860	20910134	0.10086	Aldrin	4.3
14.827	14.813	4037720	20403420	0.09786	Heptachlor Epoxide	5.1
16.076	16.062	3550888	19093284	0.09903	Endosulfan I	5.3
16.844	16.831	3493138	18120846	0.10378	4,4'-DDE	5.1
17.013	17.000	3505292	19276548	0.10149	Dieldrin	5.3
17.853	17.840	2850720	15899954	0.10073	Endrin	5.3
18.200	18.187	2970576	17255298	0.10058	Endosulfan II	5.9
18.476	18.467	2558561	14317632	0.10534	4,4'-DDD	5.3
18.947	18.933	2293562	14369134	0.09870	Endrin Aldehyde	5.9
	19.994	4916625	28954380	0.20205	Endosulfan Sulfate/4,4'-DDT	
19.924	19.994	2537691	15111502	0.10612	- C1	5.9A
20.022	19.994	2378934	13842878	0.09733	- C1	6.4A
21.924	21.911	2828633	17980842	0.10123	Endrin Ketone	6.1
22.556	22.542	1320037	8544393	0.09932	Methoxychlor	6.4
24.071	24.058	1982278	13492099	0.10227	Dibutylchlorendate (DBC)	6.4
28.787	28.773	894407	5311870	0.00000	Octachloronapthalene	6.1
29.831	29.822	2404065	17219860	0.09531	Decachlorobiphenyl (DCB)	6.9

Totals			
Unknowns	1711055	8916265	N/A
Quantified	76549920	363012000	2.09842
Grand Total	78260976	371928256	2.09842

MISSING PEAKS

No missing peaks.

PEAK GROUP INFORMATION

No peak group information.

ANALYSIS SUMMARY

Method..... TOCP
Run sequence..... T1005
Calibration..... T1005
External standard calibration using area
Calibration last modified on 6-OCT-1992 at 08:23

Uncalibrated peaks use user factor (0.0000)

B-11A

Industrial OCP/PCB Target Compound Linear Ranges

Compound	Instrument Levels Low Point	Instrument Levels High Point
Tetrachlro-m-xylene(TCX)	0.005ug/mL	0.100ug/mL
alpha-BHC	0.005ug/mL	0.100ug/mL
gamma-BHC	0.005ug/mL	0.100ug/mL
beta-BHC	0.005ug/mL	0.100ug/mL
Heptachlor	0.005ug/mL	0.100ug/mL
delta-BHC	0.005ug/mL	0.100ug/mL
Aldrin	0.005ug/mL	0.100ug/mL
Heptachlor Epoxide	0.005ug/mL	0.100ug/mL
Endosulfan I	0.005ug/mL	0.100ug/mL
4,4'-DDD	0.005ug/mL	0.100ug/mL
Endosulfan II	0.005ug/mL	0.100ug/mL
4,4'-DDT	0.005ug/mL	0.100ug/mL
Endrin Aldehyde	0.005ug/mL	0.100ug/mL
Endosulfan II	0.005ug/mL	0.100ug/mL
Dibutylchlorendate(DBC)	0.005ug/mL	0.100ug/mL
Methoxychlor	0.005ug/mL	0.100ug/mL
Endrin Ketone	0.005ug/mL	0.100ug/mL
Decachlorobiphenyl(DCB)	0.005ug/mL	0.100ug/mL
Alpha Chlordane	0.005ug/mL	0.100ug/mL
Gamma Chlordane	0.005ug/mL	0.100ug/mL
Technical Chlordane	0.075ug/mL*	
Toxaphene	0.250ug/mL	1.50ug/mL
Aroclor 1221	0.05ug/mL	0.45ug/mL
Aroclor 1232	0.05ug/mL	0.45ug/mL
Aroclor 1242	0.05ug/mL	0.45ug/mL
Aroclor 1248	0.05ug/mL	0.45ug/mL
Aroclor 1254	0.05ug/mL	0.45ug/mL
Aroclor 1260	0.05ug/mL	0.45ug/mL
Diallate	0.100ug/mL*	
Isodrin	0.005ug/mL	0.100ug/mL
Chlorobenzilate	0.100ug/mL*	
Kepone	0.250ug/mL*	
Mirex	0.005ug/mL	0.100ug/mL
Hexachloropentadiene(HCCPD)	0.005ug/mL	0.100ug/mL

* Linear range for this compound has not been demonstrated. The running level standard concentration has been listed. Analysts should dilute samples within +/- 2.0X of this standard.