ARV

MALCOLM PIRNIE

Draft

Corrective Measures Monitoring Plan

Building 40 Bedrock Groundwater
Main Manufacturing Area
Watervliet Arsenal
Watervliet, New York

Baltimore Corps of Engineers
Baltimore, Maryland

Prepared by:

Malcolm Pirnie, Inc.
43 British American Blvd.
Latham, New York 12110

US Army Corps of Engineers

August 2004
0285934

DEPARTMENT OF THE ARMY
WATERVLIET ARSENAL
1 Buffington Street
WATERVLIET, NY 12189-4000
August 12, 2004

REPLY TO
ATTENTION OF:

Facilities Engineering

Ms. Renee Gelblat
RCRA Project Manager
RCRA Programs Branch
U.S. Environmental Protection Agency
290 Broadway, 22nd Floor
New York, New York 10007

Re: Corrective Measures Monitoring Plan
Building 40
Main Manufacturing Area, Watervliet Arsenal, Watervliet, New York

Dear Ms. Gelblat:

Enclosed please find the draft Corrective Measures Monitoring Plan (CMMP) for the bedrock groundwater corrective measures at Building 40 of the Watervliet Arsenal, Watervliet, New York (WVA). The CMMP has been prepared based on the various discussions with the New York State Department of Environmental Conservation (NYSDEC) and United States Environmental Protection Agency (USEPA) from November 2003 through June 2004, and in accordance with the comments provided by the agencies on the October 2003 Draft Corrective Measures Work Plan.

As you are aware, the WVA and the U.S. Army are committed to initiating the first permanganate injection by September 30, 2004. As such, we request an expedited review of this draft CMMP, if possible. We apologize for any inconvenience this may cause and appreciate your help as we endeavor to meet our target schedule. In the meantime, we are available to meet and/or discuss the draft CMMP at your convenience. Please contact JoAnn Kellogg of the WVA at (518) 266-5296, or Stephen Wood of the USACE at (410) 962-3506 if you have any questions concerning this matter.

THOMAS E. POND
Acting Chief, Facilities Engineering

Attachments

NIH ISO 9002 REGISTERED COMPANY ANSI-RAB QMS ACCREDITED

Copies Furnished:

Tony R. Perry (Environmental Scientist)
U.S.Army Environmental Center
Environmental Restoration Division (ERD), Bldg 4480
Aberdeen Proving Ground
Aberdeen, Maryland 21010-5401
(1 copy)

Ms. Charlotte M. Bethoney
Bureau of Environmental Exposure Investigation
Division of Environmental Health Assessment
New York State Department of Health
Flanigan Square
547 River Street, Room 300
Troy, NY 12180
(1 copy)

Commander, U.S. Army Corps of Engineers, Baltimore District
10 South Howard Street, Baltimore, MD 21201
ATTN: Mr. Steve Wood
(2 copies)

New York State Department of Environmental Conservation
Bureau of Radiation & Hazardous Site Management-8th Floor
625 Broadway-Albany, N.Y. 12233-7050
ATTN: Mr. Larry Rosenmann and Mr. Victor Valaitis
(1 copy each)

New York State Department of Environmental Conservation
Region 4
RCRA Programs Branch
ATTN: Mr. Cliff VanGelder
1150 N Westcott Road
Schenectady, N.Y. 12306-2014
(1 copy)

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF APPENDICES

1.0 INTRODUCTION AND PURPOSE

On behalf of the Watervliet Arsenal (WVA), Malcolm Pirnie, Inc. (Malcolm Pirnie) is conducting a Corrective Measures (CM) program for the bedrock groundwater at Building 40 of the WVA, which is located in the City of Watervliet, New York (Figure 1-1). The CM program is being conducted under contract with the U.S. Army Corps of Engineers (USACE), Baltimore District in accordance with an Administrative Order on Consent between the WVA, the New York State Department of Environmental Conservation (NYSDEC), and the United States Environmental Protection Agency (USEPA). Details for the CM program are presented in the draft *Corrective Measures Work Plan, Building 40 Bedrock Groundwater, Main Manufacturing Area, Watervliet Arsenal, Watervliet, New York* (Malcolm Pirnie, 2004) (CM Work Plan).

The CM program is designed to treat the chlorinated volatile organic compounds (CVOCs), composed primarily of tetrachloroethene (PCE), trichloroethene (TCE), cis-1,2-dichloroethene (cDCE), and, to a lesser extent, vinyl chloride (VC), that are present in the bedrock groundwater and shale bedrock matrix at Building 40. This treatment will be accomplished using injections of sodium and potassium permanganate (herein collectively referred to as "permanganate") throughout the treatment area.

1.1 PURPOSE

The purpose of this Corrective Measures Monitoring Plan (CMMP) is to present the groundwater monitoring requirements and procedures to be conducted during the permanganate injection and rebound monitoring periods of the corrective measures. The CMMP details the type, location, and construction of multi-level monitoring wells to be installed to monitor the corrective measures; sampling and analytical methods; sampling frequency; and quality assurance/quality control (QA/QC) procedures. The CMMP also includes information collected during drilling and subsequent borehole geophysical characterization of the monitoring well boreholes installed as part of the CM Work Plan.

2.0 MONITORING WELL INSTALLATION TESTING RESULTS

In accordance with the CM Work Plan, borehole testing was conducted during and after the installation of six bedrock boundary monitoring wells and two upgradient bedrock monitoring wells to evaluate groundwater flow characteristics and the vertical distribution of CVOCs in the bedrock groundwater. The results of this testing are presented below. Testing locations are shown on the site map presented in Figure 2-1.

2.1 DISCRETE INTERVAL PACKER SAMPLING

Discrete interval packer testing was conducted in June and July 2004 during the drilling of monitoring wells MW-89 and MW-90, which are located in the northwestern portion of Building 40. The discrete interval of each borehole was purged until three volumes had been removed. A groundwater sample was then collected for VOC analysis under a rapid turnaround time. Pumping rates and water levels in the discrete interval were monitored during purging to evaluate the relative hydraulic properties of each borehole interval. If an interval was pumped dry, the borehole was allowed to recharge, then pumped dry again, before groundwater samples were collected. Discrete interval packer testing results are shown in Table 2-1. Analytical laboratory reporting forms for the packer testing samples are presented in Appendix A.

As shown in Table 2-1, relatively low concentrations of CVOCs were detected in the samples collected from monitoring well MW-90, which is located in the courtyard between Unit 5 and Unit 6 of Building 40. As discussed in the CM Work Plan, the presumed source of the CVOCs is in the area of MW-90. CVOCs were not detected in the interval from 59 to 79 feet below ground surface (bgs) at monitoring well MW-89. Sampling intervals above this depth did not contain water. Given the results at MW-90, and the fact that MW-89 is located upgradient of MW-90, monitoring well MW-89 was not advanced farther.

2.2 BOREHOLE HYDROGEOPHYSICAL TESTING

Borehole geophysical testing was conducted in boundary monitoring wells MW-81, MW-82R, MW-83, MW-84R, MW-85R, and MW-86R (see Figure 2-1) upon the completion of well installation and development in early July 2004. The objectives of the geophysical characterization were to:

- Evaluate groundwater flow parameters (i.e., transmissivity) in each boundary well

borehole and in each of the anticipated monitoring zones; and

- Identify the depth and nature (i.e., width and relative flow) of major fractures intersecting each boundary well borehole.

The primary corrective measures performance objectives are to reduce the concentration and associated groundwater mass flux of CVOCs in the groundwater at the compliance boundary. As discussed in subsequent sections, changes in CVOC concentration and groundwater mass flux will be evaluated through sampling of depth-discrete monitoring zones in each of the compliance boundary monitoring wells. Accordingly, the geophysical evaluation was designed to assess groundwater flow parameters (i.e., degree of hydraulic connection and transmissivity) in each of the compliance boundary monitoring wells. A secondary objective of the geophysical investigation was to identify the depth and nature (i.e., aperture and dip) of major fractures intersecting the borehole so that monitoring zones could be adjusted accordingly and to allow for better assessment of the distribution of permanganate during the corrective measures.

The following tests were performed in each boundary monitoring well:

- Gamma Ray;
- Spontaneous Potential (SP);
- Single Point Resistance (SPR);
- Short and long normal Resistivity (MW-81 and MW-83);
- 3-Arm Caliper;
- Fluid Temperature;
- Fluid Resistivity;
- Acoustic Televiewer
- Optical Televiewer (OBI);
- Full Waveform Sonic; and
- Heat pulse flow meter under ambient and pumping conditions.

During logging, fractures were identified in each borehole based on the combination of results from the various instruments. Single borehole heat pulse flow meter testing was then conducted to evaluate flow at each of the identified fractures and in the borehole as a whole. Limited cross borehole testing was then conducted at select wells to evaluate connectivity. The results of the geophysical testing are summarized in Table 2-2. Detailed geophysical testing results for each well are presented in Appendix B. Transmissivity calculations were performed using the

same methods (United States Geological Survey (USGS) flow modeling code FWRAP) as those used previously at the WVA (see CM Work Plan and/or *Characterization of Fractures and Flow Zones in a Contaminated Shale at the Watervliet Arsenal, Albany County, New York: USGS Open File Report 01-385* (Williams and Paillet, 2002)). As shown in Table 2-2, several fractures transmitting groundwater flow were found in each borehole; however, there are two highly transmissive fracture zones that are present along the compliance boundary. These fracture zones appear to be nearly planar sub-parallel features comprised of interconnected fractures dipping in many directions and with widths ranging from less than one foot to greater than several feet. Both features dip to the east and plunge to the north. The first feature, which was identified during previous testing at the WVA, intersects monitoring well MW-86R (94 feet bgs), MW-85R (77 feet bgs), MW-84R (49 feet bgs), and possibly, MW-83 (~36 feet bgs). This zone also appears to intersect MW-59 (91 feet bgs), MW-71 (65 feet bgs) and MW-34 (24 feet bgs). The second feature intersects MW-83 (121 feet bgs), MW-82R (94 feet bgs), and MW-81 (78 feet bgs), and probably in MW-65 (111 feet bgs). Cross-borehole testing shows that there is a connection between these two fractures features; however, the nature of this connection could not be identified during the testing.

3.0 MULTI-LEVEL MONITORING WELLS

As discussed in the CM Work Plan, the compliance monitoring array will consist of the six bedrock compliance boundary monitoring wells located along the eastern WVA property boundary. Each of these wells will be completed with a three-zone multi-level monitoring system. Achievement of the corrective action performance criteria will be based on sampling results from the 18 monitoring zones within the six property boundary monitoring wells.

3.1 MONITORING INTERVALS

The proposed monitoring intervals are presented in Table 3-1 and shown on Plate 1. The monitoring intervals are designed to evaluate the 'shallow', 'intermediate', and 'deep' portions of the compliance boundary from the bedrock surface to 150 feet bgs. As shown on Plate 1, the monitoring wells intervals have been adjusted slightly, where necessary, based on the presence of fractures identified during the geophysical profiling.

3.2 MONITORING WELL CONSTRUCTION AND DEVELOPMENT

Prior to installation of the multi-level monitoring wells, groundwater elevations in all of the fully-penetrating open borehole wells at Building 40 will be measured to evaluate the direction of groundwater flow in the area. Each multi-level monitoring well will be completed with three nested wells using standard well installation techniques. Each well will be constructed with one-inch inner diameter (I.D.) PVC pipe and 0.020-inch slot screen. The annular space around each screen will be backfilled with an appropriately-sized clean silica sand filter pack. The screen/filter pack interval for each well will encompass the monitoring intervals shown in Table 3-1. Each monitoring interval will be isolated through the emplacement of five feet of bentonite chips or pellets.

Upon completion, each monitoring zone will be equipped with a dedicated bladder or positive-displacement pump and developed to remove groundwater present prior to multilevel well construction. At a minimum, each well will be developed until three monitoring zone interval volumes have been purged from the well. Field parameters, including turbidity, pH, temperature, and specific conductivity, will be measured during development and recorded on a well development log. Purged groundwater will be temporarily contained in a polyethylene tank pending characterization and off-site disposal/treatment at a permitted facility in accordance with local, state, and federal regulations.

4.0 SAMPLING LOCATIONS AND FREQUENCY

The Building 40 Corrective Measures monitoring locations are presented in Table 4-1. This table also indicates monitoring frequency and parameters that will be monitored during the implementation of the corrective measures. Sampling methodology is discussed in Sections 5.0 and 6.0. Modifications to the sampling program may be necessary based on the results of the permanganate injections. Any such modifications will be approved by the NYSDEC and USEPA prior to implementation.

4.1 BASELINE MONITORING

Two baseline monitoring events will be conducted prior to the first permanganate injection event at the locations presented in Figure 4-1. The second monitoring event will be conducted approximately two weeks after the completion of the first monitoring event. Groundwater samples from each well will be analyzed for VOCs to establish baseline concentrations from which to evaluate treatment efficacy. In addition to VOC analyses, samples from each well will also be analyzed for potassium, sodium, sulfate, specific conductivity, and pH to allow for the evaluation of changes in groundwater chemistry during the injection program. The average concentration of each of the parameters analyzed during the two baseline monitoring events will be used as the baseline concentration.

4.2 PERMANGANATE INJECTION MONITORING

Permanganate injection monitoring will be conducted during the five-year permanganate injection period and will consist of two tasks: Pre-Injection Monitoring and Permanganate Distribution Monitoring. Details for these tasks are presented below.

4.2.1 Pre-Injection Monitoring

Pre-injection monitoring will be conducted prior to each permanganate injection event at the same locations sampled during the baseline monitoring. Each sample will be analyzed for the same parameters as the baseline samples (see Table 4-1). Prior to the collection of samples, groundwater removed from each well during purging will be assessed for the presence and concentration of permanganate. If permanganate is present in the well at concentrations greater than 1,000 ppm, no samples will be collected for field/laboratory analysis. If permanganate is not present in the well, or

is present at a concentration less than 1,000 ppm, then groundwater samples will be collected for field/laboratory analysis.

4.2.2 Permanganate Distribution Monitoring

Permanganate distribution monitoring will be conducted during all phases (Phases I, II, and III – see CM Work Plan) of the five-year permanganate injection program at the six compliance boundary monitoring wells. The initial monitoring frequency will be monthly; however, sampling frequency may be subsequently reduced based on permanganate persistence. In addition, during Phase I of the permanganate injection program, distribution monitoring will also be conducted at injection wells IW-1 through IW-4 and wells MW-79 and MW-88. Distribution monitoring at wells MW-79 and MW-88 will be stopped once Phase I injections are initiated at well MW-79. Permanganate presence, permanganate concentration, and specific conductance will be evaluated during each distribution monitoring event. The presence of permanganate will be based on the presence or absence of permanganate's characteristic purple color in the purged groundwater. Permanganate concentrations will be measured in the field using a photospectrometer. Specific conductance will also be measured in the field using a specific conductivity meter.

4.3 REBOUND MONITORING

Rebound monitoring will begin after the completion of the five-year permanganate injection program and after permanganate is no longer present in one or more of the compliance boundary monitoring wells. Rebound monitoring will be conducted quarterly for the first year of sampling, then semi-annually for the remaining four years. Monitoring locations and sample analyses will be the same as those used for the baseline sampling (see Table 4-1). Prior to the collection of samples, groundwater removed from each well during purging will be assessed for the presence and concentration of permanganate. If permanganate is present in the well at concentrations greater than 1,000 ppm, no samples will be collected for field/laboratory analysis. If permanganate is not present in the well, or is present at a concentration less than 1,000 ppm, then groundwater samples will be collected for field/laboratory analysis.

5.0 FIELD SAMPLING

5.1 SAMPLING METHODOLOGY

Groundwater sampling methodology will be the same for baseline, permanganate injection, and rebound monitoring. Groundwater in each monitoring well or monitoring zone will be purged prior to sample collection using low-flow purging procedures. A maximum of one well or monitoring interval volume will be removed from each monitoring well prior to sampling using a bladder, positive displacement, or submersible pump. This methodology will minimize any borehole storage effects that may affect permanganate distribution monitoring, minimize the amount of permanganate removed from the subsurface during sampling, and will avoid significant changes to the natural fracture flow system in each borehole. Detailed procedures for monitoring well purging and sampling are presented in the Quality Assurance Project Plan (QAPP) in Section 6.0.

5.2 INVESTIGATION DERIVED WASTE

Purge water resulting from monitoring well sampling activities will be contained and disposed in accordance with local, state, and federal regulations.

6.0 QUALITY ASSURANCE PROJECT PLAN (QAPP)

6.1 INTRODUCTION

The objective of this Quality Assurance Project Plan (QAPP) is to ensure that data produced from the corrective measures monitoring is of sufficient quality and quantity to measure the success of the Building 40 Corrective Measures. To meet this objective, the following six topics are presented and discussed in this section:

- Project organization and responsibilities
- Data quality objectives and analytical requirements
- Sample collection procedures
- Sample integrity
- Field data collection procedures
- Field instrument calibration and maintenance

Samples collected during the performance of the corrective measures will be analyzed by Severn Trent Laboratories, Inc. (STL), a commercial laboratory. Field measurements and analyses will be performed by Malcolm Pirnie staff.

6.2 PROJECT ORGANIZATION AND RESPONSIBILITIES

Malcolm Pirnie will conduct or supervise all engineering and field operations. An organizational chart for the project is presented on Figure 6-1.

The Malcolm Pirnie Project Officer is the representative with contract authority. The Project Officer is responsible for appointing the Project Manager, supervising staff in the performance of project duties, and providing corporate support. The Project Manager, in turn, is responsible for managing the project staff and communicating with the USACE, assuring that all project quality control (QC) procedures are followed.

The Analytical Laboratory Manager is responsible for the overall management of the analytical laboratory, including the appointment and supervision of departmental managers, and for approving all analytical procedures and associated QC procedures.

The Laboratory quality assurance/quality control (QA/QC) Supervisor acts as liaison between Malcolm Pirnie field and laboratory operations and is responsible for:

1. Ensuring that field personnel are familiar with and adhere to proper sampling procedures,

field measurement techniques, sample identification, and chain-of custody procedures.

2. Coordination of sample analyses to meet project objectives.
3. Preparations of analytical reports, including coordination with the Project Manager to assure that the data are validated prior to release outside of the analytical laboratory.
4. Review of laboratory data for compliance with precision, accuracy, and completeness objectives.
5. Review of any QC deficiencies reported by the Analytical Laboratory Manager.
6. Coordination of any data changes resulting from review by the Project Manager.

The Project Manager and Laboratory QA/QC Supervisor are responsible for providing consistent and accurate field or laboratory data and technical reports produced by analysts, project scientists or engineers, and sampling personnel under their supervision. These individuals are responsible to the Project Officer for ensuring that all personnel under their direction are knowledgeable of the QA/QC requirements of the project and that all QC and technical review procedures are followed, and documentation is provided.

It is the responsibility of all project personnel, as well as the laboratory analysts, project scientists, and field team members, to perform and document the required QA/QC procedures. It is the responsibility of laboratory analysts to perform preliminary QC checks to ensure that each batch of data being generated passes all required QC criteria. Field team members must bring any unusual observations or analytical problems to the immediate attention of the Project Manager.

6.3 DATA QUALITY OBJECTIVES

The overall data quality objective is to ensure that data of known and acceptable quality are generated. The quality of data is measured through qualitative and quantitative parameters, known collectively as the PARCCS parameters (Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity). All analytical work shall be conducted using laboratory validated procedures.

- **Precision** is a measure of mutual agreement among individual measurements of the same property, usually under prescribed conditions. Assessing precision measures the random error component of the data collection process. Precision is determined by measuring the agreement among individual measurements of the same property, under similar conditions. The degree of agreement, expressed as the relative percent difference (RPD),

is calculated using the formula below.

$$\text{RPD} = \frac{(V_1 - V_2)}{\frac{(V_1 + V_2)}{2}} \times 100$$

where: V1 = value 1; V2 = value 2

Analytical precision is assessed by analyzing matrix spike/matrix spike duplicate pairs and laboratory duplicate samples. Field precision is assessed by measurement of field duplicate samples. The objective for precision is to equal or exceed the precision demonstrated for similar samples and should be with the established control limits for the methods. Precision control limits and QC RPD limits can be found in the laboratory's SOP for each analytical parameter.

- **Accuracy** is the degree of agreement of a measurement with an accepted reference or true value. Accuracy measures the bias or systematic error of the entire data collection process. Sources of these errors include the sampling process, field and laboratory contamination, sample preservation and handling, sample matrix interferences, sample preparation methods, and calibration and analytical procedures. To determine accuracy, a reference material of known concentration is analyzed or a sample that has been spiked with a known concentration is reanalyzed. Accuracy is expressed as a percent recovery and is calculated using the following formula:

$$\%\ \text{Recovery} = 100 \times \frac{\text{measured value}}{\text{true value}}$$

Recoveries are assessed to determine method efficiency and matrix interference effects. Analytical accuracy is measured by the analysis of calibration checks, system blanks, quality control samples, surrogate spikes, matrix spikes, and other checks required by the selected analytical methods. Sampling accuracy is assessed by evaluating the results of filtration and trip blanks. Sampling accuracy is also maintained by frequent and thorough review of field procedures. The objective is to meet or exceed the demonstrated accuracy for the analytical methods on similar samples and should be

within established control limits for the methods. Accuracy control limits and MS/MSD and surrogate recovery limits are located within each laboratory SOP, where applicable.

- **Representativeness** expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is achieved through proper development of the sampling plan. The sampling plan must be designed so that the samples collected are as representative as possible of the medium being sampled and that a sufficient number of samples will be collected.

- **Completeness** is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Data is complete and valid if it meets all acceptance criteria including accuracy, precision, and any other criteria specified by the particular analytical method being used. Completeness is calculated as follows:

  % Completeness=100 x V/n

  where: V = number of measurements judged valid

  n = total number of measurements

  The objective is to generate a sufficient database with which to make informed decisions. To help meet the completeness objective, every effort must be made to avoid sample loss through accidents or inadvertence. The completeness goal for field collection is 100%. The completeness goal for laboratory analysis is 95%. The Project Manager has the responsibility of deciding whether re-sampling and re-analysis are required to meet the data quality objectives. The Project Manager will discuss the situation with the project team and then inform the Laboratory QA/QC Supervisor of the decision.

- **Comparability** expresses the confidence with which one data set can be compared to another. Comparability cannot be described in quantitative terms, but must be considered in designing the sampling program. Thus, this objective will be met by using standard methods for sampling and analyses and by following techniques and methods set forth in this Work Plan.

- **Sensitivity** is a measure of a method's detection limit and ability to distinguish between two values. Sensitivity is determined through the laboratory's Instrument Detection Limit (IDL) and Minimum Detection Limit (MDL) studies. The DL for the analytical parameters have been reviewed and satisfy the program objectives.

Proper execution of each project task is needed to yield consistent information, results that are representative of the media and conditions being measured, and data that are useful for meeting the intended project objectives.

The analytical laboratory will perform the analyses for specified compounds using standardized methods, thereby generating data to provide a baseline for establishing control limits (for precision, accuracy, reporting limits) for daily analyses.

6.4 LABORATORY ANALYTICAL PROCEDURES

Laboratory analyses will be conducted by STL. Permanganate concentration will be analyzed using field instrumentation. Standard operating procedures (SOPs) for STL are provided in Appendix C.

6.5 SAMPLE COLLECTION PROCEDURES

The sampling procedures described in this plan are designed to ensure collection of representative samples for analysis, and are based on the following sources:

a. USEPA Region II Groundwater Sampling Procedure, Low Stress (Low Flow) Purging and Sampling, March, 1998.
b. USEPA Region II CERCLA Quality Assurance Manual, October, 1989.
c. NYS Department of Environmental Conservation Analytical Services Protocol 9/89, Revisions 12/91, and any subsequent modifications.
d. RCRA Quality Assurance Project Plan Guidance, NYS Department of Environmental Conservation, Division of Hazardous Substances Regulation, 3/29/91.
e. USEPA RCRA Ground Water Monitoring Technical Enforcement Guidance Document, September 1986.

The objectives for each field team member are to:

- Collect a sample that is representative of the matrix being sampled.
- Maintain sample integrity from the time of sample collection to receipt by the laboratory.

6.5.1 Decontamination of Sampling Equipment

Cross contamination of samples from any source is to be avoided. All sampling equipment must be clean and free from the residue of any previous samples. To accomplish this, the following procedures will be followed:

- All non-dedicated sampling equipment must be cleaned initially and prior to being reused. The following is the procedure for decontamination.
- Wash and scrub with low phosphate detergent.
- Rinse with tap water.
- Rinse with ten percent nitric acid if metals analysis is required.
- Rinse with tap water.
- Rinse with isopropanol (pesticide grade).
- Rinse thoroughly with analyte-free deionized water.
- Air dry.
- Wrap in aluminum foil for transport.
- To decontaminate non-dedicated sampling pumps, the following procedure will be followed before and after each well is sampled:
    - Pre-rinse: Operate pump in a deep basin containing 8 to 10 gallons of potable water for 5 minutes.
    - Wash: Operate pump in a deep basin containing 8 to 10 gallons of a non-phosphate detergent solution, such as Alconox, for 5 minutes. Use the detergent sparingly.
    - Rinse: Operate pump in a deep basin of potable water for 5 minutes.
    - Final Rinse: Operate pump in a deep basin of distilled/deionized water to pump out 1 to 2 gallons of this final rinse water.

Field measurement equipment, such as pH and conductivity meters will be rinsed prior to and after each use with analyte-free deionized water.

6.5.2 Sampling Equipment

The following equipment will be needed to collect groundwater samples for laboratory

analysis and to perform field analyses:

- Electric water level indicator
- Bladder pump, positive displacement pump, or submersible pump
- Air compressor
- Generator
- Polyethylene discharge tubing
- pH meter
- Specific Conductivity Meter
- Turbidity meter
- Photoionization Detector (PID)
- Field logbook and field logs
- Laboratory prepared sample containers
- Roll of polyethylene sheeting
- Decontamination equipment

6.5.3 Sampling Procedures

Groundwater sampling will be conducted in accordance with the USEPA Low-Flow Sampling Protocol (USEPA 1998). A piece of polyethylene sheeting will be fitted over the monitoring well and laid on the ground. The sampling equipment will be placed on the polyethylene sheeting. The expansion cap will be removed and the headspace at the top of the monitoring well will be measured with a PID. This step may be omitted in those monitoring wells which have already demonstrated in the previous rounds of water level measurement that they contain no or insignificant amounts of vapors or gases. The PID will be calibrated before the start of each sampling event.

The well will be purged at a rate suitable to minimize drawdown. Field parameters, consisting of pH, specific conductance, temperature, turbidity, and water level will be measured in each monitoring well prior to, during, and after purging (just before sampling). Both the pH and the specific conductivity meters will be calibrated for water temperature before each sampling event.

The volume of water removed from each monitoring well will be dependent upon the amount of time required for stabilization of the field parameters. However, as discussed in Section 5.1, a maximum of one well or monitoring interval volume will be removed from each monitoring well

prior to sampling to minimize the amount of permanganate removed from the subsurface during sampling. In general, the well will be considered stabilized for sample collection when field parameters have stabilized for three consecutive readings as follows:

- pH: +/- 0.1 standard units
- Specific Conductance: +/- 3%
- Turbidity +/- 10%

When the field parameters have stabilized, the volume of water purged will be recorded, and groundwater in the monitoring well will be sampled through the pump at the same flow rate used to purge the well. If permanganate is present in the purge water at the initiation of sampling, or if permanganate is observed in the purge water during purging, then monitoring of field parameters will be discontinued and the monitoring well will be purged of one well of monitoring interval volume, at the same flow rate, prior to sampling.

The sample bottles will be pre-preserved by the laboratory. The preservation requirements are presented on Table 6-1. The sample bottles will be immediately placed in a cooler held at 4°C. If, upon the completion of purging, there is no visible evidence of permanganate in the purged groundwater (i.e., purple color), three 40-ml vials will be filled, without leaving any headspace, for analysis of CVOCs by STL using USEPA Method 8260B. If visible permanganate is present in the purged groundwater, a sample will be collected for analysis of permanganate concentration using a field photospectrometer. If the permanganate concentration in the sample is less than 1,000 ppm, the permanganate in the sample will be quenched by adding a solution of sodium bisulfite, then sent STL for analysis of VOCs by USEPA Method 8260B.

6.5.4 Field Documentation

Data to be recorded in the field logbook will include the date, start and finish times, purging and sampling methods, depth to water, volume of water removed during purging, sample depth interval, field parameters, field-analyzed chemical measurements and PID readings. In addition, the field technician shall note the weather during sampling, samples taken, instrument maintenance and calibration, and any field changes, problems or corrective actions.

6.5.5 Containers, Preservatives, and Holding Times

Sample integrity is preserved through the use of proper sample containers, addition of the correct preservatives to the samples, and meeting designated holding times (the time from sample

collection to sample analysis). The field team leader is responsible for proper sample collection, labeling, preservation, and shipment to the laboratory to meet required holding times. Table 6-1 identifies the proper containers, preservation techniques, and maximum holding times.

The analytical laboratory will supply Malcolm Pirnie with commercially-cleaned sample containers. The containers will meet or exceed cleaning and quality control requirements of USEPA OSWER Directive 9240.0-05, Specifications for Obtaining Contaminant-Free Sample Containers. Sample containers will be stored in clean, dust-free areas that are segregated from the analytical laboratory and solvent/reagent storage areas.

6.5.6 Quality Control Samples

QA/QC samples in the form of trip blanks, blind duplicates, matrix spikes (MS), and matrix spike duplicates (MSD) will be collected to assess field and laboratory accuracy and precision techniques. The collection procedures and frequency of collection of these samples are presented below.

6.5.6.1 Trip Blanks

When collecting environmental aqueous samples for volatile organic compound analysis, a trip blank is taken into the field as part of the sampling kit (the set of appropriate containers used to collect the samples). Trip blanks consist of demonstrated analyte-free water sealed in 40 ml Teflon®-lined septum vials. These blanks are used to determine whether collected samples have been contaminated by outside sources during shipment or storage. Trip blanks will be prepared by STL and carried with every shipment of aqueous samples that are to be analyzed for volatile organic compounds.

6.5.6.2 Field duplicates

Field duplicates are collected in such a manner that they are equally representative of parameters of interest at a given point in space and time. They are separate from laboratory duplicates, which demonstrate analytical precision. Field duplicates will be collected at a rate of one per 20 environmental samples. The field duplicate samples will be "blind" duplicates, meaning that the laboratory must not know that the sample is a duplicate; therefore, the duplicates will be identified in the same manner as the other samples, with a fictitious sample location. The duplicate samples will be identified in the field notes, but not on the chain-of-custody form recorded by the

field team at the time of collection.

6.5.6.3 Matrix Spike/Matrix Spike Duplicates (MS/MSD)

MS and MSD samples will be collected at three times their standard volume at the frequency of one per 20 environmental samples. This will provide the laboratory with the required additional volume for performing QC analysis on-site specific samples. The additional sample volume is spiked with a known quantity and quality of the appropriate analytes. The percent recovery will be used to calculate accuracy. The relative percent difference (RPD) for each component will be used to calculate precision.

6.5.7 Data Validation

The laboratory will be responsible for validating their results in accordance with the supplied SOPs attached in Appendix A. Third-party data validation will be performed for baseline, pre-injection, and rebound monitoring events as described in Section 7.1.

6.5.8 Sample Custody

An essential part of any program that requires sampling and analysis is ensuring sample integrity from collection to data reporting. This includes the ability to trace the possession and handling of samples from collection through analysis and final disposition. The documentation of the history of the sample is referred to as chain-of-custody. This section addresses the following sample custody procedures:

- Sample Identification and Labeling
- Sample Custody in the Field
- Sample Shipping
- Sample Custody in the Laboratory
- Document Control

6.5.8.1 Sample Identification and Labeling

Affixing a unique sample label to each sample container will identify all samples collected. Indelible ink will be used to complete sample labels. After they are affixed to the containers, the labels will be covered with clear plastic waterproof tape.

Each sample will have a unique designation, using alphanumeric codes that will identify the

site, the type of sample, the sample location, and the series number at the location. The codes to be used and examples of sample designations using the codes are provided below. The labels will not indicate that a sample is a duplicate or a blank.

CODES:

WVA	Watervliet Arsenal
(50-100)	Depth Interval (if multi-level monitoring well)

Examples:

WVA-MW-78 (50-100)

Each label will contain the following information:

1. Site Name
2. Project Number
3. Sample Number
4. Sample Matrix
5. Company Name
6. Parameters to be Analyzed
7. Date of Collection
8. Time of Collection
9. Preservation Technique Employed
10. Sampler's Name

6.5.8.2 Sample Custody in the Field

Sample custody in the field consists of documenting all field activities related to sampling and establishing an accurate written record that traces the possession and handling of each sample from the moment of its collection, through shipment to the laboratory, and ultimately through analysis. The custody procedures described herein conform to US Army Corps of Engineers Guidance ER 1100-1-263, Chemical Data Quality Management for Hazardous Waste Remedial Activities, and are modeled after standard USEPA procedures.

Field activities will be documented in a field notebook. All field notes will be recorded in indelible ink on standard forms or in bound notebooks. All standard forms used during the field

investigation will be bound in a notebook and centrally located on-site at the end of each day. The notebook will be signed and dated at the end of each day. Similarly, significant events occurring during the day will be reported to the project manager at the end of each day.

At a minimum, the notebook will contain the following sample particulars:

- Sample number
- Date and time of sample collection
- Sample location
- Name of collector
- Analytical work to be done
- Type of sample, and whether the sample is a duplicate, quality assurance, or quality control sample
- Volume of sample taken
- Type of container, number of containers/samples
- Any field observations or measurements (e.g. pH, temperature, specific conductance)
- Type of concentration: low, medium, high
- Preservatives used
- Sampling methodology/special features
- Sampler's signature
- Method of shipment to the laboratory

After samples are collected, chain-of-custody records will be used to trace the possession and handling of the samples. A chain-of-custody record is a printed form that accompanies a sample or group of samples as custody is transferred from person to person.

As soon as practical after sample collection, the following information must be entered, in indelible ink, on the chain-of-custody record:

f. Project number.

g. Project name.

h. Sampler(s) signature(s).

i. Sample identification code for each sample contained in the shipment. This code appears on the sample label.

j. The date-of-collection of each sample, entered as six-digit number indicating the year, month, and day.

k. The time-of-collection of each sample, entered as a four-digit number indicating the military time of collection; for example, the time entered for a sample collected at 1:54 p.m. would be 13:54 hrs.

l. The matrix of each sample (e.g. soil, aqueous, sludge).

m. The analysis and analytical method to be performed for each sample.

n. The number of containers for each sample identification code (when analyzing for several chemical parameters, a number of containers are filled at each sampling location).

o. Remarks. Enter any appropriate remarks.

A person is in custody of a sample if the sample is:

- In that person's physical possession;
- In view after being in that person's physical possession;
- Placed in a locked repository by that person, or;
- Placed in a secure, restricted area by that person.

Custody of the samples may be transferred several times prior to their arrival at the laboratory. For example, a field team shipper may be designated to receive all samples from field team members. When transferring custody to another responsible individual, perform the following:

1. Enter the date and time of sample transfer on the chain-of-custody form, and sign the form, under the "Relinquished by:" entry.
2. Make certain that the individual receiving custody signs the "Received by:" entry.

When transferring custody to a common carrier (e.g. Federal Express), perform the following:

1. Enter the date and time of sample transfer on the chain-of-custody form, and sign the form, under the "Relinquished by:" entry.
2. Enter the name of the carrier under the "Received by:" entry.
3. Enter the bill-of-lading or Federal Express air bill number under the "Remarks:" entry.
4. Follow the packaging procedures presented in Section 6.7.3 (Sampling Shipping)

6.5.9 Sample Shipping

The following procedures shall be followed for packaging and shipping of samples:

- Coolers shall be used to ship samples.
- All labels shall be written with indelible ink.
- Approximately 3 inches of inert cushioning material such as vermiculite shall be placed in the bottom of the cooler.
- Each sample container shall be enclosed in a clear plastic bag through which the labels are visible, and the bag sealed. The containers shall be placed upright in the cooler in such a way that they do not touch, and will not touch during shipping.
- Additional vermiculite packing material shall be placed in the cooler to partially cover the sample containers (more than halfway). Bags of ice shall then be placed around, among, and on top of the sample containers.
- The cooler shall then be filled with cushioning material.
- The original chain-of-custody form shall be placed in a waterproof plastic bag and placed inside the cooler. Retain a copy of the form with the field records.
- The drain of the cooler shall be taped shut.
- The cooler lid shall be secured by taping. The cooler shall be wrapped completely with strapping tape at a minimum of two locations in such a way that no labels are covered.
- The shipping label shall be attached to top of cooler.
- "This Side Up" labels with arrows and "Fragile" labels shall be placed on at least two sides of the cooler.
- Numbered and signed custody seals shall be affixed on the front right and back left sides of the cooler, across the lid and body of the cooler. These seals shall be covered with wide, clear tape.

6.5.10 Sample Custody in the Laboratory

Once the samples arrive at the laboratory, custody of the samples will be maintained by laboratory personnel. Each sample will be identified upon receipt by the laboratory and cross-referenced to the chain-of-custody record. Any inconsistencies will be noted on the custody record. Laboratory personnel will immediately notify the Malcolm Pirnie Laboratory QA/QC Supervisor, Site Field Manager, or Project Manager if inconsistencies are identified.

The analytical laboratory will have written SOPs for maintaining security of samples and tracking the work performed on samples through the entire analytical process. The SOP requires that sample receipt, sample extraction/preparation, sample analysis, data reduction and data reporting be documented by the laboratory.

6.5.11 Document Control

Document control consists of maintaining a project file, an analytical laboratory batch file, a project field file, and a QA project file. The project file will be maintained by the Malcolm Pirnie Project Manager and will contain all original documents. Project personnel may keep their own files; however, all original documents will be kept in the project file. All laboratory records, including batch forms, log sheets, and computerized worksheets, will be kept by the analytical laboratory in a batch file in the sample control center. Field logs will be maintained by the Project Manager in a project field file. The Laboratory QA/QC Supervisor will independently maintain a QA project file. At the end of the project, the QA project file will be turned over to the Project Manager. The following documents will be placed in the QA project file:

1. QA records maintained throughout the investigation.
2. Documentation of QA system and performance audits.
3. Documentation of all unusual findings or observations.
4. Documentation of all QA corrective actions.
5. All official QA correspondence received or issued relating to the investigation, including records of telephone calls.
6. One copy of all QA deliverable review sheets.
7. Any other QA documents related to the project or follow-up activities related to the investigation.

6.5.12 Equipment Calibration and Maintenance Procedures

Instruments must be properly calibrated to produce technically valid data. Documented calibration and calibration check results verify that the instruments used for measurement are in proper working order and the data produced is reliable. When calibration requirements are met, the data will support the focused investigation decisions dealing with the nature and extent of contamination and safety concerns. In the event that the data is used in court, documented calibrations are necessary to ensure that the data is legally defensible.

6.5.13 Laboratory Calibration Procedures

All samples shall be analyzed according to the analytical methods attached in Appendix C, and shall follow the procedures described by the methods. All calibration results shall be recorded and kept on file, and will be reviewed and evaluated by the data validator as part of analytical data validation procedures.

Instrument calibration will be checked with a reference standard prior to the analysis of any sample. The standards used for calibrations will be traceable and each calibration will be recorded in the laboratory notebook for the particular analysis. Any printouts, chromatograms, etc., generated for the calibration will be kept on file.

6.5.14 Corrective Actions

Corrective actions are those measures taken to rectify a laboratory or field measurement system that does not comply with this QAPP. The need for corrective action may be identified by system or performance audits or by standard QC procedures. The essential steps in the corrective action system are:

1. Identifying and defining the problem.
2. Assigning of responsibility for investigating the problem.
3. Investigating and determining the cause of the problem.
4. Determining a corrective action to eliminate the problem.
5. Assigning and accepting responsibility for implementing the corrective action.
6. Implementing the corrective action and evaluating its effectiveness.
7. Verifying that the corrective action has eliminated the problem.

A nonconformance is defined as an identified or suspected deficiency in an approved document (e.g., technical report, analysis, calculation, computer program); an item where the quality of the end item itself or subsequent activities using the document or item would be affected by the deficiency; or an activity that is not conducted in accordance with the established plans or procedures. Any staff member engaged in project work who discovers or suspects a nonconformance is responsible for initiating a nonconformance report. The Laboratory QA/QC Supervisor shall evaluate each nonconformance report and shall provide a disposition, which describes the actions to be taken. The Project Manager shall ensure that no further project work dependent on the nonconforming item or activity is performed until approval is obtained and the nonconformance

report is closed out. If the nonconformance is related to material, the Project Manager shall be responsible for marking or identifying, with the nonconformance report number, the nonconforming item (if practical) and indicating that it is nonconforming and is not to be used.

Samples that are analyzed prior to the resolution of a nonconforming event will be resampled if project staff are still in the field, and/or reanalyzed once the corrective action has been demonstrated to be effective.

A copy of each closed nonconformance report shall be included in the quality assurance file. Copies of all nonconformances shall be maintained by the Laboratory QA/QC Supervisor.

6.5.15 Field Corrective Actions

At the end of each sampling day, the sampling team shall report any problems requiring corrective action that were encountered during the day. Corrective action will be undertaken when a non-conforming condition is identified. A non-conforming condition occurs when QA objectives for precision, accuracy, completeness, representativeness or comparability are not met, or when procedural practices or other conditions are not acceptable. A report shall be filed which documents the problems encountered and the corrective action implemented. Corrective actions for field test kits and instrumentation involve ordering back up kits to be available. A stop-work order may be issued by the Laboratory QA/QC Supervisor, upon authorization by the Project Manager, if corrective action does not adequately address a problem, or if no resolution can be reached.

6.5.16 Laboratory Corrective Actions

If a particular analysis is deemed "out of control," corrective action will be taken to ensure continued data quality. Actions that may be taken include, but are not limited to:

- Rechecking calculations
- Checking QC data on other samples
- Auditing laboratory procedures
- Reanalyzing the sample if the holding time requirements have not been exceeded
- Accepting data with the acknowledged level of uncertainty
- Discarding data

The coordinator of the laboratory's analytical section will be responsible for initiating laboratory corrective action when necessary. Recommendations for corrective actions outside the laboratory will be made by the Laboratory QA/QC Supervisor to the Project Manager.

7.0 REPORTING

7.1 DATA DELIVERABLES AND VALIDATION

All samples analyzed by the off-site analytical laboratory will be reported under a standard 30-day turnaround time with NYSDEC Category B deliverables. Third-party data validation will be performed for 10 percent of the results from off-site laboratory-analyzed samples. Data validation results will be presented in a Data Usability Summary Report (DUSR).

7.2 REPORTS

The following documents will be submitted as part of the Corrective Measures Monitoring Program.

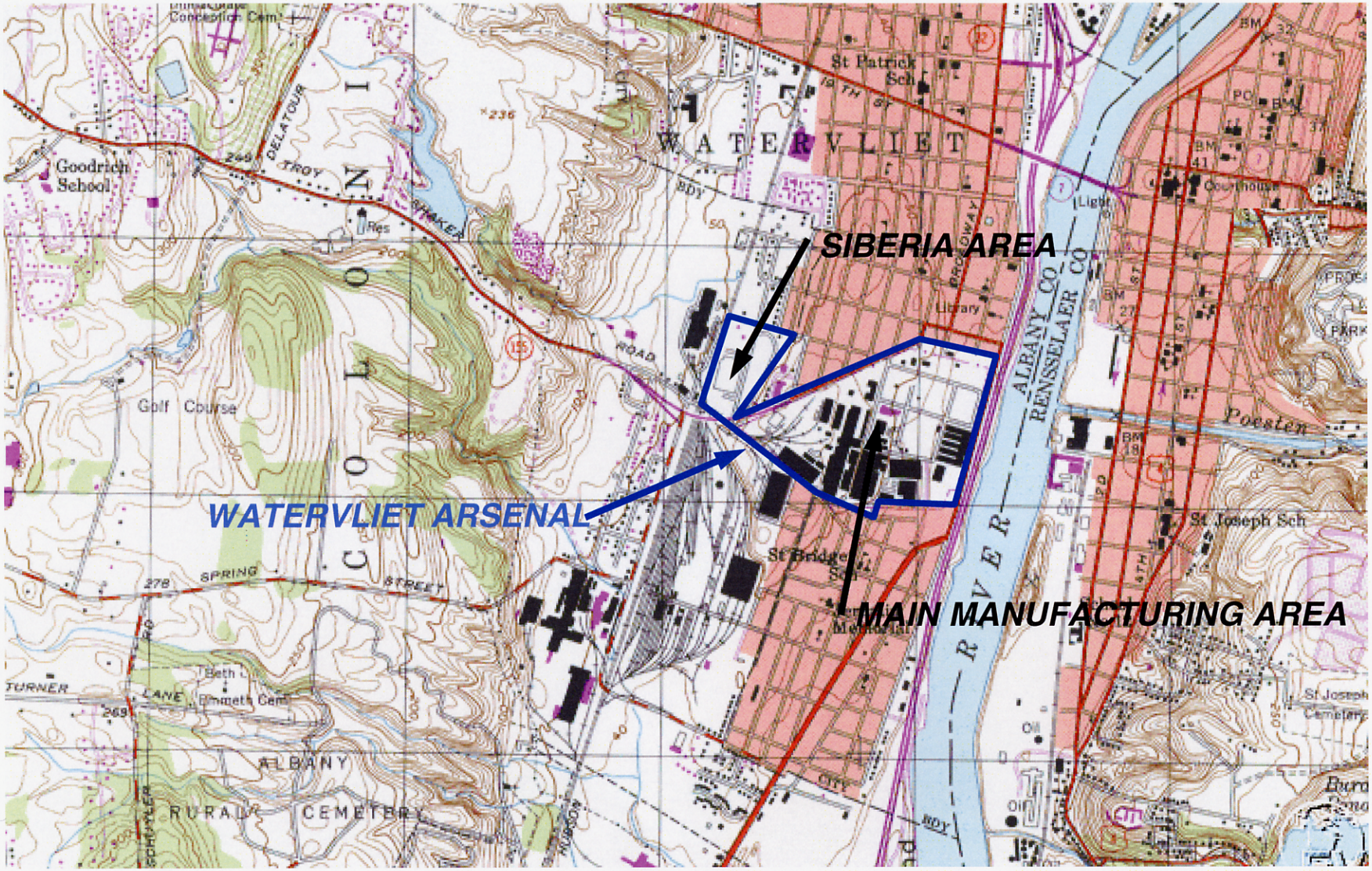
- **CM Startup Report**: As discussed in the CM Work Plan, a CM startup report will be submitted upon the completion and evaluation of the first Phase I permanganate injection. Results for the two baseline monitoring events will be presented in the Startup Report.
- **Pre-Injection Monitoring Summary Reports**: Pre-injection monitoring summary reports will be submitted upon the receipt, validation, and review of the analytical data from each pre-injection monitoring event. The reports will include the results of the monthly monitoring conducted between injections.
- **Rebound Monitoring Summary Reports**: Rebound monitoring summary reports will be submitted upon the receipt, validation, and review of the analytical data from each rebound monitoring event.

8.0 REFERENCES

Malcolm Pirnie, Inc., 2004. *Draft Corrective Measures Work Plan, Building 40 Bedrock Groundwater, Main Manufacturing Area, Watervliet Arsenal, Watervliet, New York.*

USEPA, 1998, Region II Low Stress (Low-Flow) Purging and Sampling Procedure for Collecting Ground Water Samples from Monitoring Wells, Final.

Williams, J.H. and F. Paillet, 2002. *Characterization of Fractures and Flow Zones in a Contaminated Shale at the Watervliet Arsenal, Albany County, New York. (USGS Open-File Report 01-385)* [Online]. Available: http://water.usgs.gov/ogw/bgas/publications/OFR-01-385/.



SOURCE: U.S.G.S 7.5 MIN. TROY SOUTH QUADRANGLE

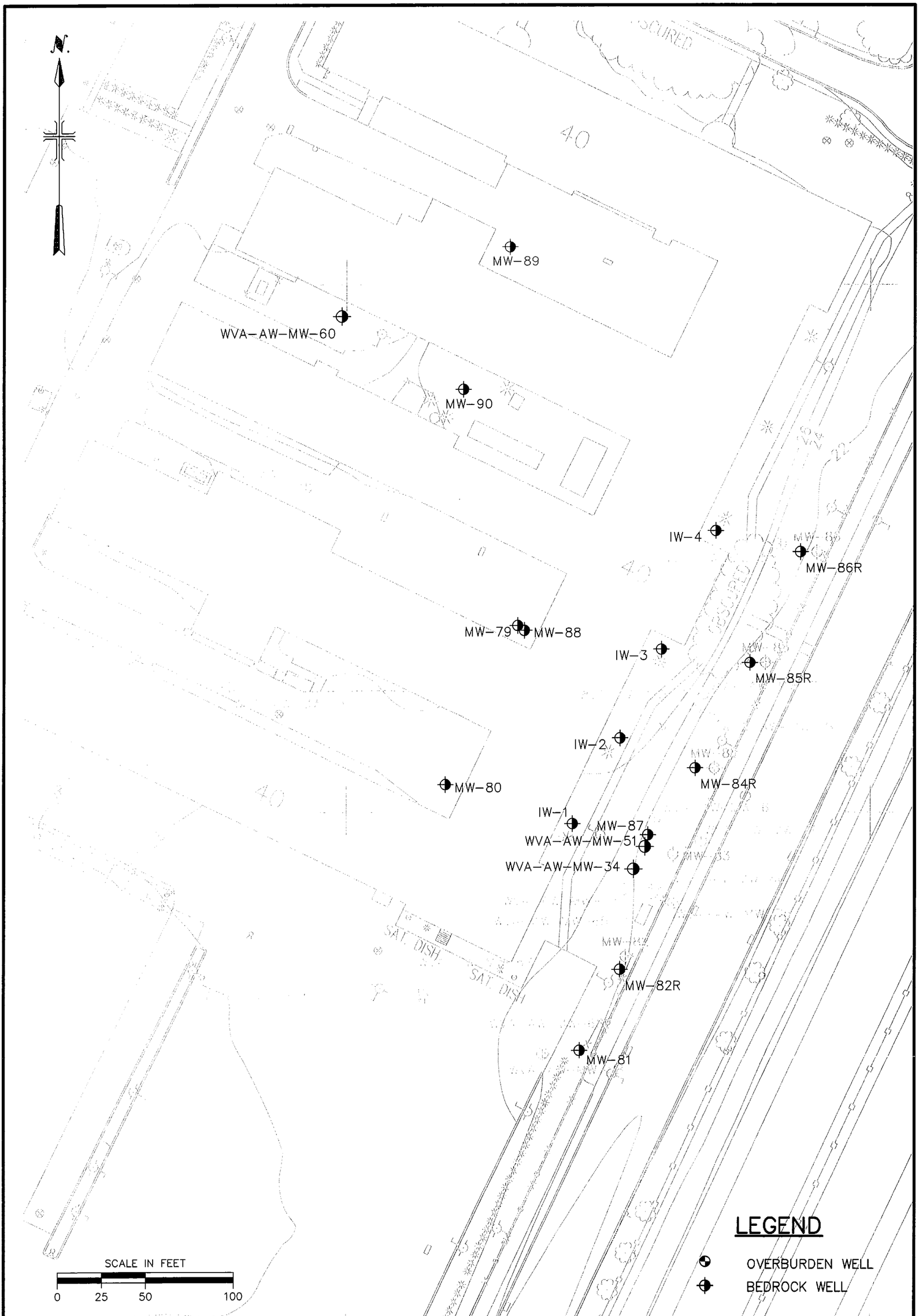
US Army Corps of Engineers
Baltimore District

SITE LOCATION

WATERVLIET ARSENAL,
WATERVLIET, NEW YORK

FIGURE 1-1

XREFS: I: \ACAD\PROJ\0285\X-REFS\BASEMAP.dwg I: \ACAD\PROJ\0285\X-REFS\WELLS-NEW.dwg IMAGES: None
User: Tyo Spec: COLONIE File: I: \ACAD\PROJ\0285\939\CMMP FIG. 2-1.DWG Scale: 1:1 Date: 08/12/2004 Time: 11:47 Layout: Layout1

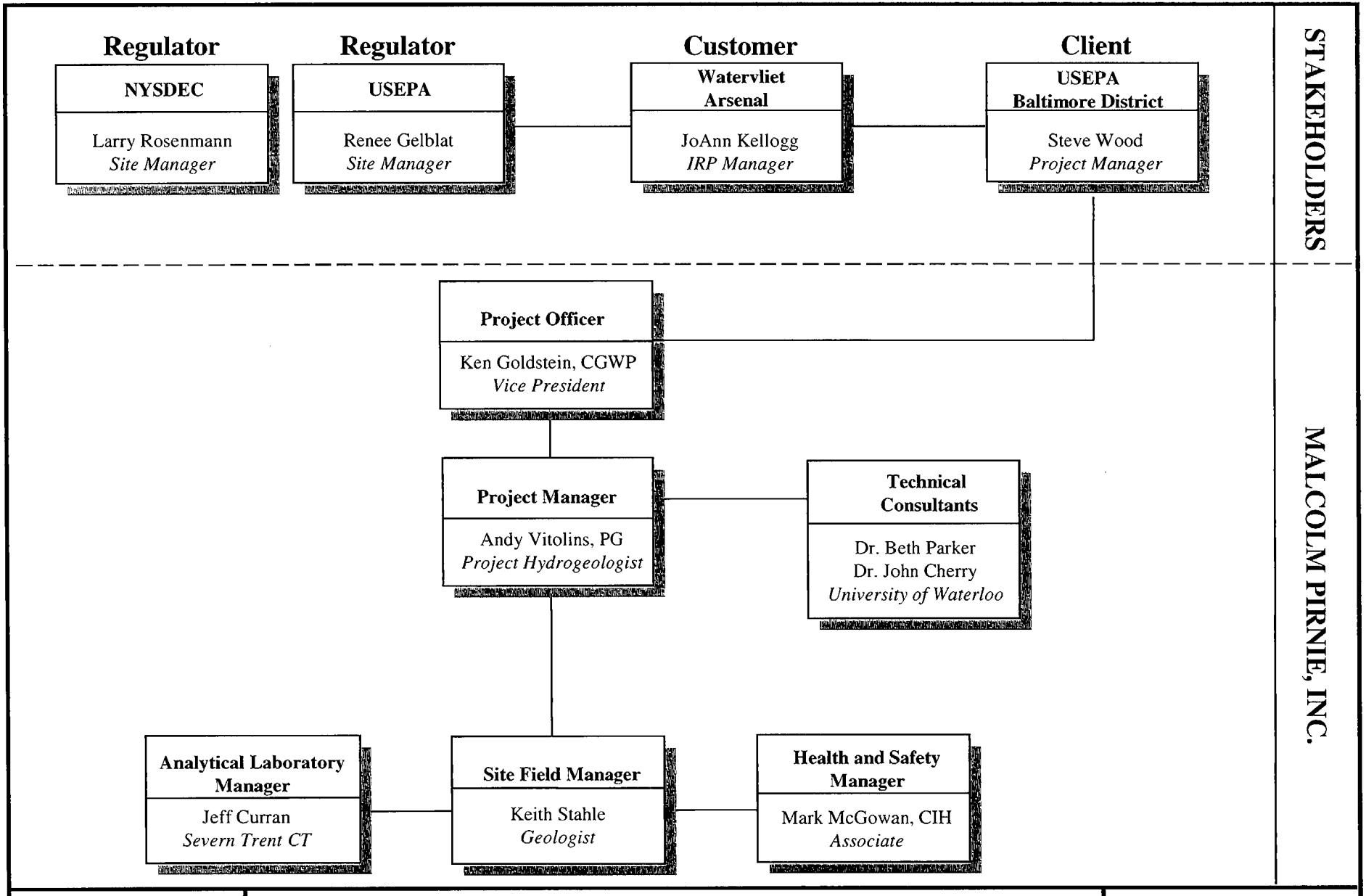


MALCOLM PIRNIE

WATERVLIET ARSENAL
BUILDING 40 CORRECTIVE MEASURES
SITE MAP

COPYRIGHT © 2004
MALCOLM PIRNIE, INC.

FIGURE 2-1



US Army Corps of Engineers
Baltimore District

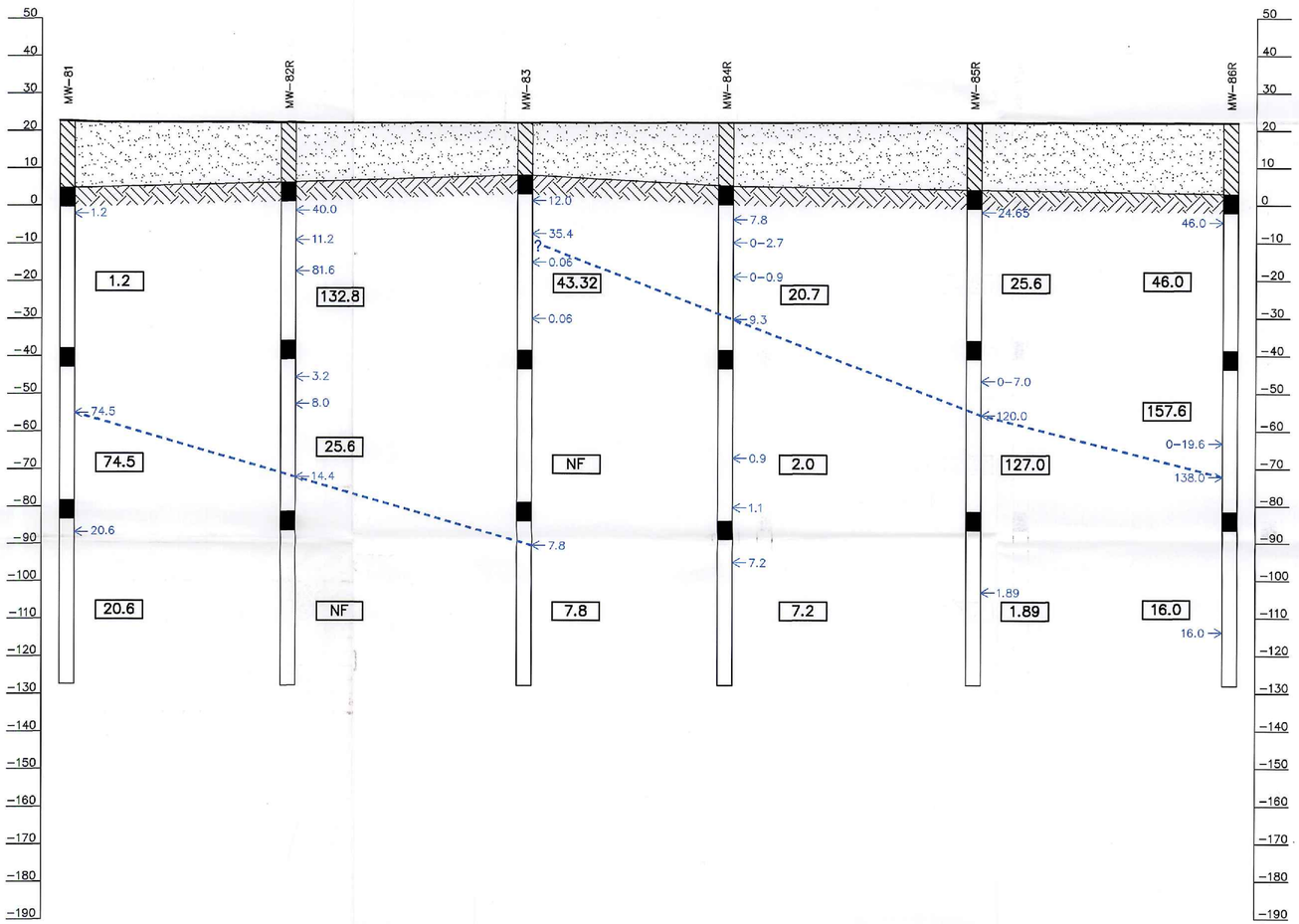
PROJECT ORGANIZATION

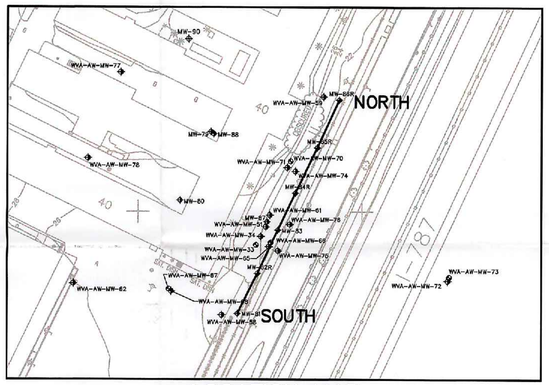
BUILDING 40 GROUNDWATER CORRECTIVE MEASURES
WATERVLIET ARSENAL, WATERVLIET, NEW YORK

FIGURE 6-1

SOUTH

NORTH





LOCATION MAP

SCALE: 1" = 100'

LEGEND

- OVERBURDEN
- COMPETENT BEDROCK SURFACE
- 1.2 — FRACTURE W/FLOW (TRANSMISSIVITY FT²/D)
- 74.5 — MONITORING ZONE TRANSMISSIVITY (FT²/D)
- NF — NO FLOW DETECTED
- FRACTURE WITH KNOWN CONNECTION
- CASED INTERVAL
- BENTONITE SEAL
- SCREENED INTERVAL

10 0 10 20

SCALE: 1" = 20'

WARNING – IT IS A VIOLATION OF NEW YORK EDUCATION LAW SECTION 7209.2, FOR ANY PERSON, UNLESS HE IS ACTING UNDER THE DIRECTION OF A LICENSED PROFESSIONAL ENGINEER OR LAND SURVEYOR, TO ALTER THIS DOCUMENT IN ANY WAY. IF ALTERED THE ALTERING PERSON SHALL COMPLY WITH THE REQUIREMENTS OF NEW YORK EDUCATION LAW, SECTION 7209.2.

MALCOLM PIRNIE

REVISIONS

NO.	BY	DATE	REMARKS

DES _____

DWN _____

CKD _____

WATERVLIET ARSENAL

BUILDING 40 BEDROCK CORRECTIVE MEASURES

MULTI-LEVEL BOUNDARY MONITORING WELL CONSTRUCTION

SCALE: 1" = 20'

PLATE 1

XREFS:I:\ACAD\PROJ\0285\X-REFS\BASEMAP.dwg I:\ACAD\PROJ\0285\X-REFS\WELLS-NEW.dwg IMAGES:None
User:Tyo Spec:COLONIE Files:I:\ACAD\PROJ\0285\639\CROSS-SECT.DWG Scale:1:1 Date:08/04/2004 Time:08:52 Layout:Layout2

Table 2-1
Summary of Groundwater Sample Results
Target Volatile Organic Compounds
June 2004 Discrete Interval Sampling
Watervliet Arsenal, Watervliet, New York

Well ID	Sample Interval (Feet)	Concentration (ug/l)				
		Vinyl Chloride	c-1,2 DCE	TCE	PCE	Total
MW-89	19-39	Dry				
	39-59	Dry				
	59-79					0
MW-90	18-38					0
	38-58		150		7	157
	58-78		40		11	51
	78-98		19		5	24
	98-150	120	170			290

Notes:
Blank cell - analyte not detected

Table 2-2
Summary of Borehole Fractures and Flow
Compliance Boundary Monitoring Wells
Building 40 Corrective Measures
Watervliet Arsenal, Watervliet, New York

Monitoring Well	Depth (feet bgs)	Elevation (feet AMSL)	Transmissivity (ft^2/d)	
			Pumping Rate 1	Pumping Rate 2
MW-81			***0.59 gpm***	***1.05 gpm***
	24	-4.28	1.20	0.60
	78.2	-58.48	74.50	72.00
	109.4	-89.68	20.60	19.20
MW-82R			***0.91 gpm***	***NT***
	23.44	-3.98	40.00	
	30.53	-11.07	11.20	
	37.77	-18.31	81.60	
	67.09	-47.63	3.20	
	75.22	-55.76	8.00	
	93.9	-74.44	14.40	
MW-83			***0.91 gpm***	***NT***
	19.26	-0.52	12.00	
	25.08	-6.34	35.40	
	32.77	-14.03	0.06	
	50.76	-32.02	0.06	
	111.27	-92.53	7.80	
MW-84R			***0.51 gpm***	***0.91 gpm***
	25.52	-6.79	7.80	6.30
	29.89	-11.16	NF	2.70
	40.14	-21.41	NF	0.90
	49.01	-30.28	9.30	7.20
	91.11	-72.38	0.90	1.80
	101.71	-82.98	1.10	0.45
	118.16	-99.43	7.20	3.45
MW-85R			***0.59 gpm***	***1.0 gpm***
	23.64	-4.84	24.65	25.20
	69.15	-50.35	NF	7.00
	77.01	-58.21	120.35	103.60
	125.48	-106.68	1.89	0.98
MW-86R			***0.48 gpm***	***1.05 gpm***
	24.94	-5.81	46.00	49.00
	85.91	-66.78	NF	19.60
	94.68	-75.55	138.00	126.00
	135.59	-116.46	16.00	8.40

Notes:
bgs - below ground surface
ft^2/d - square feet per day
gpm - gallons per minute
NT - not tested
NF - no flow detected
Fractures with documented connection.
AMSL - above mean sea level

Table 3-1
Summary of Multi-level Monitoring Well Construction
Compliance Boundary Monitoring Wells
Building 40 Corrective Measures
Watervliet Arsenal, Watervliet, New York

Monitoring Well	Casing Depth (feet bgs)	Description	Interval (feet bgs)	Length of Interval (feet)
MW-81	23	Zone 1 Bentonite Seal	18-23	5
		Monitoring Zone 1	23-60	37
		Zone 2 Bentonite Seal	60-65	5
		Monitoring Zone 2	65-101	36
		Zone 3 Bentonite Seal	101-106	5
		Monitoring Zone 3	106-150	44
MW-82R	21	Zone 1 Bentonite Seal	16-21	5
		Monitoring Zone 1	21-58	37
		Zone 2 Bentonite Seal	58-63	5
		Monitoring Zone 2	63-103	40
		Zone 3 Bentonite Seal	103-108	5
		Monitoring Zone 3	108-150	42
MW-83	19	Zone 1 Bentonite Seal	14-19	5
		Monitoring Zone 1	19-60	41
		Zone 2 Bentonite Seal	60-65	5
		Monitoring Zone 2	65-101	36
		Zone 3 Bentonite Seal	101-106	5
		Monitoring Zone 3	106-150	44
MW-84R	22	Zone 1 Bentonite Seal	17-22	5
		Monitoring Zone 1	22-60	38
		Zone 2 Bentonite Seal	60-65	5
		Monitoring Zone 2	65-106	41
		Zone 3 Bentonite Seal	106-111	5
		Monitoring Zone 3	111-150	39
MW-85R	23	Zone 1 Bentonite Seal	18-23	5
		Monitoring Zone 1	23-58	35
		Zone 2 Bentonite Seal	58-63	5
		Monitoring Zone 2	63-103	40
		Zone 3 Bentonite Seal	103-108	5
		Monitoring Zone 3	108-150	42
MW-86R	24	Zone 1 Bentonite Seal	19-24	5
		Monitoring Zone 1	24-60	36
		Zone 2 Bentonite Seal	60-65	5
		Monitoring Zone 2	65-103	38
		Zone 3 Bentonite Seal	103-108	5
		Monitoring Zone 3	108-150	42

Notes:
bgs - below ground surface

Table 4-1
Groundwater Monitoring Summary
Building 40 Corrective Measures
Watervliet Arsenal, Watervliet, New York

Phase	Wells Monitored	Frequency	Analyses (Method)
Baseline Monitoring	MW-81* MW-82R* MW-83* MW-84R* MW-85R* MW-86R* MW-34 MW-51 MW-60 MW-80 MW-87 MW-89	2 events	VOCs (USEPA 8260B) Sulfate (USEPA 300.0) Potassium (USEPA 6010B) Sodium (USEPA 6010B) Specific Conductance (Field) pH (Field) Turbidity (Field)
Pre-Injection Monitoring	MW-81* MW-82R* MW-83* MW-84R* MW-85R* MW-86R* MW-34 MW-51 MW-60 MW-80 MW-87 MW-89	Prior to Injection	Permanganate (visual) Permanganate (spectrometer) VOCs (USEPA 8260B)** Sulfate (USEPA 300.0)** Potassium (USEPA 6010B)** Sodium (USEPA 6010B)** Specific Conductance (Field)** pH (Field)** Turbidity (Field)**
Permanganate Distribution Monitoring	MW-81* MW-82R* MW-83* MW-84R* MW-85R* MW-86R* MW-79*** MW-88*** IW-1*** IW-2*** IW-3*** IW-4***	Monthly during 5-year injection period. May be reduced based on permanganate persistence.	Permanganate (visual) Permanganate (spectrometer)

Notes:
* Multi-level well (all zones sampled)
** To be analyzed only if permanganate concentration < 1,000 ppm in monitoring well/interval.
*** Monitored during Phase I injections only.

Table 4-1
Groundwater Monitoring Summary
Building 40 Corrective Measures
Watervliet Arsenal, Watervliet, New York

Phase	Wells Monitored	Frequency	Analyses (Method)
Rebound Monitoring	MW-81* MW-82R* MW-83* MW-84R* MW-85R* MW-86R* MW-34 MW-51 MW-60 MW-80 MW-87 MW-89	Quarterly (Year 1) Semi-annual (Years 2-5)	Permanganate (visual) Permanganate (spectrometer) VOCs (USEPA 8260B)** Sulfate (USEPA 300.0)** Potassium (USEPA 6010B)** Sodium (USEPA 6010B)** Specific Conductance (Field)** pH (Field)** Turbidity (Field)**

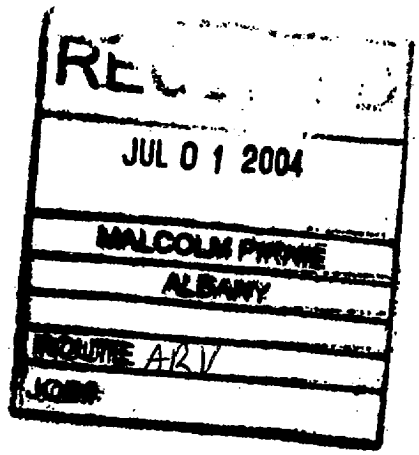
Notes:
* Multi-level well (all zones sampled)
** To be analyzed only if permanganate concentration < 1,000 ppm in monitoring well/interval.
*** Monitored during Phase I injections only.

Table 6-1
Sample Container, Preservation, and Holding Time Requirements
Building 40 Corrective Measures Monitoring
Watervliet Arsenal, Watervliet, New York

ANALYSIS	CONTAINER	PRESERVATION	HOLDING TIME
VOCs by USEPA 8260B	2 - 40 ml VOA vile w/ septum cap	HCl to pH<2; Cool to 4 deg. C	7 days
Sulfate by USEPA 300.0	1 - 500 ml plastic	Cool to 4 deg. C	28 days
Potassium by USEPA 6010B	1 - 1L plastic	HNO_3 to pH<2; Cool to 4 deg. C	180 days
Sodium by USEPA 6010B	1 - 1L plastic	HNO_3 to pH<2; Cool to 4 deg. C	180 days

Notes:
ml - milliliters
L - liter

Adirondack Environmental Services, Inc.®
Experience is the solution
314 North Pearl Street ♦ Albany, New York 12207
(800) 848-4983 ♦ (518) 434-4546 ♦ Fax (518) 434-0891



June 25, 2004

Andy Vitolin
Malcolm Pirnie, Inc.
15 Cornell Road
Latham, NY 12110
TEL: (518) 786-7349
FAX: (518) 786-8645

RE: WVA-BLDG 40

Order No.: 040624031

Dear Andy Vitolin:

Adirondack Environmental Services, Inc received 3 samples on 6/24/2004 for the analyses presented in the following report.

There were no problems with the analyses and all associated QC met EPA or laboratory specifications, except if noted.

If you have any questions regarding these tests results, please feel free to call.

ELAP#: 10709
AIHA#: 100307

Sincerely,

Christopher Hess
QA Manager

FAX:
Andy Vitolin

Adirondack Environmental Services, Inc

Date: *25-Jun-04*

CLIENT: Malcolm Pirnie, Inc.
Lab Order: 040624031
Project: WVA-BLDG 40
Lab ID: 040624031-001

Client Sample ID: MPI-89(59-79')
Collection Date: 6/24/2004
Matrix: GROUNDWATER

Analyses	Result	PQL	Qual	Units	DF	Date Analyzed
VOLATILE ORGANICS		**SW8260B**				Analyst: **ML**
Chloromethane	< 10	10		µg/L	1	6/24/2004 3:36:00 PM
Bromomethane	< 10	10		µg/L	1	6/24/2004 3:36:00 PM
Vinyl chloride	< 10	10		µg/L	1	6/24/2004 3:36:00 PM
Chloroethane	< 10	10		µg/L	1	6/24/2004 3:36:00 PM
Methylene chloride	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
Acetone	< 10	10		µg/L	1	6/24/2004 3:36:00 PM
Carbon disulfide	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
1,1-Dichloroethene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
1,1-Dichloroethane	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
trans-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
cis-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
Chloroform	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
1,2-Dichloroethane	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
2-Butanone	< 10	10		µg/L	1	6/24/2004 3:36:00 PM
1,1,1-Trichloroethane	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
Carbon tetrachloride	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
Bromodichloromethane	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
1,2-Dichloropropane	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
cis-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
Trichloroethene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
Dibromochloromethane	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
1,1,2-Trichloroethane	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
Benzene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
trans-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
Bromoform	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
4-Methyl-2-pentanone	< 10	10		µg/L	1	6/24/2004 3:36:00 PM
2-Hexanone	< 10	10		µg/L	1	6/24/2004 3:36:00 PM
Tetrachloroethene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
1,1,2,2-Tetrachloroethane	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
Toluene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
Chlorobenzene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
Ethylbenzene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
Styrene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
m,p-Xylene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM
o-Xylene	< 5.0	5.0		µg/L	1	6/24/2004 3:36:00 PM

Qualifiers:

ND - Not Detected at the Reporting Limit
J - Analyte detected below quanititation limits
B - Analyte detected in the associated Method Blank
* - Value exceeds Maximum Contaminant Level
S - Spike Recovery outside accepted recovery limits
R - RPD outside accepted recovery limits
E - Value above quantitation range

Adirondack Environmental Services, Inc

Date: *25-Jun-04*

CLIENT:	Malcolm Pirnie, Inc.	**Client Sample ID:**	MPI-90(18-38')
Lab Order:	040624031	**Collection Date:**	6/24/2004
Project:	WVA-BLDG 40		
Lab ID:	040624031-002	**Matrix:**	GROUNDWATER

Analyses	Result	PQL	Qual	Units	DF	Date Analyzed
VOLATILE ORGANICS		**SW8260B**				Analyst: **ML**
Chloromethane	< 10	10		µg/L	1	6/24/2004 4:05:00 PM
Bromomethane	< 10	10		µg/L	1	6/24/2004 4:05:00 PM
Vinyl chloride	< 10	10		µg/L	1	6/24/2004 4:05:00 PM
Chloroethane	< 10	10		µg/L	1	6/24/2004 4:05:00 PM
Methylene chloride	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
Acetone	< 10	10		µg/L	1	6/24/2004 4:05:00 PM
Carbon disulfide	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
1,1-Dichloroethene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
1,1-Dichloroethane	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
trans-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
cis-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
Chloroform	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
1,2-Dichloroethane	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
2-Butanone	< 10	10		µg/L	1	6/24/2004 4:05:00 PM
1,1,1-Trichloroethane	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
Carbon tetrachloride	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
Bromodichloromethane	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
1,2-Dichloropropane	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
cis-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
Trichloroethene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
Dibromochloromethane	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
1,1,2-Trichloroethane	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
Benzene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
trans-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
Bromoform	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
4-Methyl-2-pentanone	< 10	10		µg/L	1	6/24/2004 4:05:00 PM
2-Hexanone	< 10	10		µg/L	1	6/24/2004 4:05:00 PM
Tetrachloroethene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
1,1,2,2-Tetrachloroethane	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
Toluene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
Chlorobenzene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
Ethylbenzene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
Styrene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
m,p-Xylene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM
o-Xylene	< 5.0	5.0		µg/L	1	6/24/2004 4:05:00 PM

Qualifiers:

ND - Not Detected at the Reporting Limit

J - Analyte detected below quanititation limits

B - Analyte detected in the associated Method Blank

* - Value exceeds Maximum Contaminant Level

S - Spike Recovery outside accepted recovery limits

R - RPD outside accepted recovery limits

E - Value above quantitation range

Adirondack Environmental Services, Inc

Date: *25-Jun-04*

CLIENT:	Malcolm Pirnie, Inc.	**Client Sample ID:**	Trip Blank Lot#091
Lab Order:	040624031	**Collection Date:**	6/24/2004
Project:	WVA-BLDG 40		
Lab ID:	040624031-003	**Matrix:**	WATER

Analyses	Result	PQL	Qual	Units	DF	Date Analyzed
VOLATILE ORGANICS		**SW8260B**				Analyst: **ML**
Chloromethane	< 10	10		µg/L	1	6/24/2004 4:34:00 PM
Bromomethane	< 10	10		µg/L	1	6/24/2004 4:34:00 PM
Vinyl chloride	< 10	10		µg/L	1	6/24/2004 4:34:00 PM
Chloroethane	< 10	10		µg/L	1	6/24/2004 4:34:00 PM
Methylene chloride	6.8	5.0		µg/L	1	6/24/2004 4:34:00 PM
Acetone	10	10		µg/L	1	6/24/2004 4:34:00 PM
Carbon disulfide	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
1,1-Dichloroethene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
1,1-Dichloroethane	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
trans-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
cis-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
Chloroform	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
1,2-Dichloroethane	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
2-Butanone	< 10	10		µg/L	1	6/24/2004 4:34:00 PM
1,1,1-Trichloroethane	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
Carbon tetrachloride	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
Bromodichloromethane	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
1,2-Dichloropropane	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
cis-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
Trichloroethene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
Dibromochloromethane	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
1,1,2-Trichloroethane	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
Benzene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
trans-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
Bromoform	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
4-Methyl-2-pentanone	< 10	10		µg/L	1	6/24/2004 4:34:00 PM
2-Hexanone	< 10	10		µg/L	1	6/24/2004 4:34:00 PM
Tetrachloroethene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
1,1,2,2-Tetrachloroethane	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
Toluene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
Chlorobenzene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
Ethylbenzene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
Styrene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
m,p-Xylene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM
o-Xylene	< 5.0	5.0		µg/L	1	6/24/2004 4:34:00 PM

Qualifiers:

ND - Not Detected at the Reporting Limit

J - Analyte detected below quanititation limits

B - Analyte detected in the associated Method Blank

* - Value exceeds Maximum Contaminant Level

S - Spike Recovery outside accepted recovery limits

R - RPD outside accepted recovery limits

E - Value above quantitation range

Adirondack Environmental Services, Inc.®
Experience is the solution
314 North Pearl Street ♦ Albany, New York 12207
(800) 848-4983 ♦ (518) 434-4546 ♦ Fax (518) 434-0891

RECEIVED
JUL 0 1 2004
MALCOLM PIRNIE
ALBANY
LAbrecd copy
ROUTE AKV
JOB#

June 29, 2004

Andy Vitolin
Malcolm Pirnie, Inc.
43 British-American Blvd.
Latham, NY 12110
TEL: (518) 782-2139
FAX: (518) 782-0500

RE: WVA-BLDG 40

Order No.: 040628030

Dear Andy Vitolin:

Adirondack Environmental Services, Inc received 3 samples on 6/28/2004 for the analyses presented in the following report.

There were no problems with the analyses and all associated QC met EPA or laboratory specifications, except if noted.

If you have any questions regarding these tests results, please feel free to call.

Sincerely,

ELAP#: 10709
AIHA#: 100307

Christopher Hess
QA Manager

CC:
Aaron Bobar
FAX:
Andy Vitolin

Adirondack Environmental Services, Inc

Date: *29-Jun-04*

CLIENT: Malcolm Pirnie, Inc.
Lab Order: 040628030
Project: WVA-BLDG 40
Lab ID: 040628030-001

Client Sample ID: MPI-90(38-58')
Collection Date: 6/28/2004
Matrix: GROUNDWATER

Analyses	Result	PQL	Qual	Units	DF	Date Analyzed
VOLATILE ORGANICS		**SW8260B**				Analyst: **ML**
Chloromethane	< 10	10		µg/L	1	6/29/2004
Bromomethane	< 10	10		µg/L	1	6/29/2004
Vinyl chloride	< 10	10		µg/L	1	6/29/2004
Chloroethane	< 10	10		µg/L	1	6/29/2004
Methylene chloride	< 5.0	5.0		µg/L	1	6/29/2004
Acetone	< 10	10		µg/L	1	6/29/2004
Carbon disulfide	< 5.0	5.0		µg/L	1	6/29/2004
1,1-Dichloroethene	< 5.0	5.0		µg/L	1	6/29/2004
1,1-Dichloroethane	< 5.0	5.0		µg/L	1	6/29/2004
trans-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/29/2004
cis-1,2-Dichloroethene	150	5.0		µg/L	1	6/29/2004
Chloroform	< 5.0	5.0		µg/L	1	6/29/2004
1,2-Dichloroethane	< 5.0	5.0		µg/L	1	6/29/2004
2-Butanone	< 10	10		µg/L	1	6/29/2004
1,1,1-Trichloroethane	< 5.0	5.0		µg/L	1	6/29/2004
Carbon tetrachloride	< 5.0	5.0		µg/L	1	6/29/2004
Bromodichloromethane	< 5.0	5.0		µg/L	1	6/29/2004
1,2-Dichloropropane	< 5.0	5.0		µg/L	1	6/29/2004
cis-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/29/2004
Trichloroethene	< 5.0	5.0		µg/L	1	6/29/2004
Dibromochloromethane	< 5.0	5.0		µg/L	1	6/29/2004
1,1,2-Trichloroethane	< 5.0	5.0		µg/L	1	6/29/2004
Benzene	< 5.0	5.0		µg/L	1	6/29/2004
trans-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/29/2004
Bromoform	< 5.0	5.0		µg/L	1	6/29/2004
4-Methyl-2-pentanone	< 10	10		µg/L	1	6/29/2004
2-Hexanone	< 10	10		µg/L	1	6/29/2004
Tetrachloroethene	6.8	5.0		µg/L	1	6/29/2004
1,1,2,2-Tetrachloroethane	< 5.0	5.0		µg/L	1	6/29/2004
Toluene	< 5.0	5.0		µg/L	1	6/29/2004
Chlorobenzene	< 5.0	5.0		µg/L	1	6/29/2004
Ethylbenzene	< 5.0	5.0		µg/L	1	6/29/2004
Styrene	< 5.0	5.0		µg/L	1	6/29/2004
m,p-Xylene	< 5.0	5.0		µg/L	1	6/29/2004
o-Xylene	< 5.0	5.0		µg/L	1	6/29/2004

Qualifiers:

ND - Not Detected at the Reporting Limit
J - Analyte detected below quanititation limits
B - Analyte detected in the associated Method Blank
* - Value exceeds Maximum Contaminant Level
S - Spike Recovery outside accepted recovery limits
R - RPD outside accepted recovery limits
E - Value above quantitation range

Adirondack Environmental Services, Inc

Date: *29-Jun-04*

CLIENT: Malcolm Pirnie, Inc.
Lab Order: 040628030
Project: WVA-BLDG 40
Lab ID: 040628030-002

Client Sample ID: MPI-90(58-78')
Collection Date: 6/28/2004
Matrix: GROUNDWATER

Analyses	Result	PQL	Qual	Units	DF	Date Analyzed
VOLATILE ORGANICS		**SW8260B**				Analyst: **ML**
Chloromethane	< 10	10		µg/L	1	6/29/2004
Bromomethane	< 10	10		µg/L	1	6/29/2004
Vinyl chloride	< 10	10		µg/L	1	6/29/2004
Chloroethane	< 10	10		µg/L	1	6/29/2004
Methylene chloride	< 5.0	5.0		µg/L	1	6/29/2004
Acetone	15	10	B	µg/L	1	6/29/2004
Carbon disulfide	< 5.0	5.0		µg/L	1	6/29/2004
1,1-Dichloroethene	< 5.0	5.0		µg/L	1	6/29/2004
1,1-Dichloroethane	< 5.0	5.0		µg/L	1	6/29/2004
trans-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/29/2004
cis-1,2-Dichloroethene	40	5.0		µg/L	1	6/29/2004
Chloroform	< 5.0	5.0		µg/L	1	6/29/2004
1,2-Dichloroethane	< 5.0	5.0		µg/L	1	6/29/2004
2-Butanone	< 10	10		µg/L	1	6/29/2004
1,1,1-Trichloroethane	< 5.0	5.0		µg/L	1	6/29/2004
Carbon tetrachloride	< 5.0	5.0		µg/L	1	6/29/2004
Bromodichloromethane	< 5.0	5.0		µg/L	1	6/29/2004
1,2-Dichloropropane	< 5.0	5.0		µg/L	1	6/29/2004
cis-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/29/2004
Trichloroethene	< 5.0	5.0		µg/L	1	6/29/2004
Dibromochloromethane	< 5.0	5.0		µg/L	1	6/29/2004
1,1,2-Trichloroethane	< 5.0	5.0		µg/L	1	6/29/2004
Benzene	< 5.0	5.0		µg/L	1	6/29/2004
trans-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/29/2004
Bromoform	< 5.0	5.0		µg/L	1	6/29/2004
4-Methyl-2-pentanone	< 10	10		µg/L	1	6/29/2004
2-Hexanone	< 10	10		µg/L	1	6/29/2004
Tetrachloroethene	11	5.0		µg/L	1	6/29/2004
1,1,2,2-Tetrachloroethane	< 5.0	5.0		µg/L	1	6/29/2004
Toluene	< 5.0	5.0		µg/L	1	6/29/2004
Chlorobenzene	< 5.0	5.0		µg/L	1	6/29/2004
Ethylbenzene	< 5.0	5.0		µg/L	1	6/29/2004
Styrene	< 5.0	5.0		µg/L	1	6/29/2004
m,p-Xylene	< 5.0	5.0		µg/L	1	6/29/2004
o-Xylene	< 5.0	5.0		µg/L	1	6/29/2004

Qualifiers:

ND - Not Detected at the Reporting Limit
J - Analyte detected below quanititation limits
B - Analyte detected in the associated Method Blank
* - Value exceeds Maximum Contaminant Level
S - Spike Recovery outside accepted recovery limits
R - RPD outside accepted recovery limits
E - Value above quantitation range

Adirondack Environmental Services, Inc

Date: *29-Jun-04*

CLIENT:	Malcolm Pirnie, Inc.	**Client Sample ID:**	Trip Blank Lot#091
Lab Order:	040628030	**Collection Date:**	6/28/2004
Project:	WVA-BLDG 40		
Lab ID:	040628030-003	**Matrix:**	WATER

Analyses	Result	PQL	Qual	Units	DF	Date Analyzed
VOLATILE ORGANICS		**SW8260B**				Analyst: **ML**
Chloromethane	< 10	10		µg/L	1	6/29/2004
Bromomethane	< 10	10		µg/L	1	6/29/2004
Vinyl chloride	< 10	10		µg/L	1	6/29/2004
Chloroethane	< 10	10		µg/L	1	6/29/2004
Methylene chloride	< 5.0	5.0		µg/L	1	6/29/2004
Acetone	23	10	B	µg/L	1	6/29/2004
Carbon disulfide	< 5.0	5.0		µg/L	1	6/29/2004
1,1-Dichloroethene	< 5.0	5.0		µg/L	1	6/29/2004
1,1-Dichloroethane	< 5.0	5.0		µg/L	1	6/29/2004
trans-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/29/2004
cis-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/29/2004
Chloroform	< 5.0	5.0		µg/L	1	6/29/2004
1,2-Dichloroethane	< 5.0	5.0		µg/L	1	6/29/2004
2-Butanone	< 10	10		µg/L	1	6/29/2004
1,1,1-Trichloroethane	< 5.0	5.0		µg/L	1	6/29/2004
Carbon tetrachloride	< 5.0	5.0		µg/L	1	6/29/2004
Bromodichloromethane	< 5.0	5.0		µg/L	1	6/29/2004
1,2-Dichloropropane	< 5.0	5.0		µg/L	1	6/29/2004
cis-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/29/2004
Trichloroethene	< 5.0	5.0		µg/L	1	6/29/2004
Dibromochloromethane	< 5.0	5.0		µg/L	1	6/29/2004
1,1,2-Trichloroethane	< 5.0	5.0		µg/L	1	6/29/2004
Benzene	< 5.0	5.0		µg/L	1	6/29/2004
trans-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/29/2004
Bromoform	< 5.0	5.0		µg/L	1	6/29/2004
4-Methyl-2-pentanone	< 10	10		µg/L	1	6/29/2004
2-Hexanone	< 10	10		µg/L	1	6/29/2004
Tetrachloroethene	< 5.0	5.0		µg/L	1	6/29/2004
1,1,2,2-Tetrachloroethane	< 5.0	5.0		µg/L	1	6/29/2004
Toluene	< 5.0	5.0		µg/L	1	6/29/2004
Chlorobenzene	< 5.0	5.0		µg/L	1	6/29/2004
Ethylbenzene	< 5.0	5.0		µg/L	1	6/29/2004
Styrene	< 5.0	5.0		µg/L	1	6/29/2004
m,p-Xylene	< 5.0	5.0		µg/L	1	6/29/2004
o-Xylene	< 5.0	5.0		µg/L	1	6/29/2004

Qualifiers:

ND - Not Detected at the Reporting Limit

J - Analyte detected below quanititation limits

B - Analyte detected in the associated Method Blank

* - Value exceeds Maximum Contaminant Level

S - Spike Recovery outside accepted recovery limits

R - RPD outside accepted recovery limits

E - Value above quantitation range

Adirondack Environmental Services, Inc.®
Experience is the solution
314 North Pearl Street ♦ Albany, New York 12207
(800) 848-4983 ♦ (518) 434-4546 ♦ Fax (518) 434-0891

June 30, 2004

Andy Vitolin
Malcolm Pirnie, Inc.
43 British-American Blvd.
Latham, NY 12110

TEL: (518) 782-2139
FAX: (518) 782-0500

RE: WVA-BLDG 40

Order No.: 040630010

Dear Andy Vitolin:

Adirondack Environmental Services, Inc received 2 samples on 6/30/2004 for the analyses presented in the following report.

There were no problems with the analyses and all associated QC met EPA or laboratory specifications, except if noted.

If you have any questions regarding these tests results, please feel free to call.

ELAP#: 10709
AIHA#: 100307

Sincerely,

Christopher Hess
QA Manager

CC:
Aaron Bobar
FAX:
Andy Vitolin

RECEIVED
JUL 0 6 2004
MALCOLM PIRNIE
ALBANY
ROUTE
JOB# 0285939

Adirondack Environmental Services, Inc

Date: *30-Jun-04*

CLIENT: Malcolm Pirnie, Inc.
Lab Order: 040630010
Project: WVA-BLDG 40
Lab ID: 040630010-001

Client Sample ID: MPI-90(78-98)
Collection Date: 6/30/2004
Matrix: GROUNDWATER

Analyses	Result	PQL	Qual	Units	DF	Date Analyzed
VOLATILE ORGANICS		**SW8260B**				Analyst: **ML**
Chloromethane	< 10	10		µg/L	1	6/30/2004 12:44:00 PM
Bromomethane	< 10	10		µg/L	1	6/30/2004 12:44:00 PM
Vinyl chloride	< 10	10		µg/L	1	6/30/2004 12:44:00 PM
Chloroethane	< 10	10		µg/L	1	6/30/2004 12:44:00 PM
Methylene chloride	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
Acetone	19	10		µg/L	1	6/30/2004 12:44:00 PM
Carbon disulfide	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
1,1-Dichloroethene	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
1,1-Dichloroethane	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
trans-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
cis-1,2-Dichloroethene	19	5.0		µg/L	1	6/30/2004 12:44:00 PM
Chloroform	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
1,2-Dichloroethane	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
2-Butanone	< 10	10		µg/L	1	6/30/2004 12:44:00 PM
1,1,1-Trichloroethane	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
Carbon tetrachloride	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
Bromodichloromethane	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
1,2-Dichloropropane	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
cis-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
Trichloroethene	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
Dibromochloromethane	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
1,1,2-Trichloroethane	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
Benzene	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
trans-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
Bromoform	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
4-Methyl-2-pentanone	< 10	10		µg/L	1	6/30/2004 12:44:00 PM
2-Hexanone	< 10	10		µg/L	1	6/30/2004 12:44:00 PM
Tetrachloroethene	5.4	5.0		µg/L	1	6/30/2004 12:44:00 PM
1,1,2,2-Tetrachloroethane	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
Toluene	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
Chlorobenzene	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
Ethylbenzene	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
Styrene	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
m,p-Xylene	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM
o-Xylene	< 5.0	5.0		µg/L	1	6/30/2004 12:44:00 PM

Qualifiers:

ND - Not Detected at the Reporting Limit
J - Analyte detected below quanititation limits
B - Analyte detected in the associated Method Blank
* - Value exceeds Maximum Contaminant Level
S - Spike Recovery outside accepted recovery limits
R - RPD outside accepted recovery limits
E - Value above quantitation range

Adirondack Environmental Services, Inc

Date: *30-Jun-04*

CLIENT:	Malcolm Pirnie, Inc.	**Client Sample ID:**	Trip Blank Lot#091
Lab Order:	040630010	**Collection Date:**	6/30/2004
Project:	WVA-BLDG 40		
Lab ID:	040630010-002	**Matrix:**	WATER

Analyses	Result	PQL	Qual	Units	DF	Date Analyzed
VOLATILE ORGANICS		**SW8260B**				Analyst: **ML**
Chloromethane	< 10	10		µg/L	1	6/30/2004 1:13:00 PM
Bromomethane	< 10	10		µg/L	1	6/30/2004 1:13:00 PM
Vinyl chloride	< 10	10		µg/L	1	6/30/2004 1:13:00 PM
Chloroethane	< 10	10		µg/L	1	6/30/2004 1:13:00 PM
Methylene chloride	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
Acetone	< 10	10		µg/L	1	6/30/2004 1:13:00 PM
Carbon disulfide	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
1,1-Dichloroethene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
1,1-Dichloroethane	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
trans-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
cis-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
Chloroform	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
1,2-Dichloroethane	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
2-Butanone	< 10	10		µg/L	1	6/30/2004 1:13:00 PM
1,1,1-Trichloroethane	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
Carbon tetrachloride	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
Bromodichloromethane	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
1,2-Dichloropropane	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
cis-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
Trichloroethene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
Dibromochloromethane	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
1,1,2-Trichloroethane	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
Benzene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
trans-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
Bromoform	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
4-Methyl-2-pentanone	< 10	10		µg/L	1	6/30/2004 1:13:00 PM
2-Hexanone	< 10	10		µg/L	1	6/30/2004 1:13:00 PM
Tetrachloroethene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
1,1,2,2-Tetrachloroethane	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
Toluene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
Chlorobenzene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
Ethylbenzene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
Styrene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
m,p-Xylene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM
o-Xylene	< 5.0	5.0		µg/L	1	6/30/2004 1:13:00 PM

Qualifiers:

ND - Not Detected at the Reporting Limit

J - Analyte detected below quanititation limits

B - Analyte detected in the associated Method Blank

* - Value exceeds Maximum Contaminant Level

S - Spike Recovery outside accepted recovery limits

R - RPD outside accepted recovery limits

E - Value above quantitation range

Adirondack Environmental Services, Inc.®
Experience is the solution
314 North Pearl Street ♦ Albany, New York 12207
(800) 848-4983 ♦ (518) 434-4546 ♦ Fax (518) 434-0891

July 02, 2004

Andy Vitolin
Malcolm Pirnie, Inc.
43 British-American Blvd.
Latham, NY 12110
TEL: (518) 782-2139
FAX: (518) 782-0500

RE: WVA-BLDG 40 Order No.: 040701031

Dear Andy Vitolin:

Adirondack Environmental Services, Inc received 2 samples on 7/1/2004 for the analyses presented in the following report.

There were no problems with the analyses and all associated QC met EPA or laboratory specifications, except if noted.

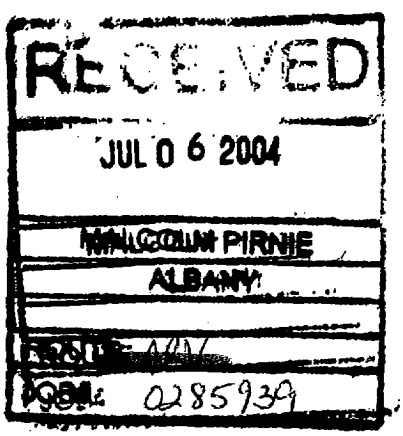
If you have any questions regarding these tests results, please feel free to call.

Sincerely,

ELAP#: 10709
AIHA#: 100307

Christopher Hess
QA Manager

FAX:
Andy Vitolin



Adirondack Environmental Services, Inc

Date: *02-Jul-04*

CLIENT: Malcolm Pirnie, Inc.
Lab Order: 040701031
Project: WVA-BLDG 40
Lab ID: 040701031-001

Client Sample ID: MPI-90(98'-150')
Collection Date: 7/1/2004
Matrix: GROUNDWATER

Analyses	Result	PQL	Qual	Units	DF	Date Analyzed
VOLATILE ORGANICS		**SW8260B**				Analyst: **ML**
Chloromethane	< 10	10		µg/L	1	7/1/2004 2:27:00 PM
Bromomethane	< 10	10		µg/L	1	7/1/2004 2:27:00 PM
Vinyl chloride	120	10		µg/L	1	7/1/2004 2:27:00 PM
Chloroethane	< 10	10		µg/L	1	7/1/2004 2:27:00 PM
Methylene chloride	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
Acetone	< 10	10		µg/L	1	7/1/2004 2:27:00 PM
Carbon disulfide	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
1,1-Dichloroethene	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
1,1-Dichloroethane	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
trans-1,2-Dichloroethene	8.1	5.0		µg/L	1	7/1/2004 2:27:00 PM
cis-1,2-Dichloroethene	170	5.0		µg/L	1	7/1/2004 2:27:00 PM
Chloroform	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
1,2-Dichloroethane	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
2-Butanone	< 10	10		µg/L	1	7/1/2004 2:27:00 PM
1,1,1-Trichloroethane	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
Carbon tetrachloride	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
Bromodichloromethane	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
1,2-Dichloropropane	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
cis-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
Trichloroethene	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
Dibromochloromethane	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
1,1,2-Trichloroethane	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
Benzene	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
trans-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
Bromoform	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
4-Methyl-2-pentanone	< 10	10		µg/L	1	7/1/2004 2:27:00 PM
2-Hexanone	< 10	10		µg/L	1	7/1/2004 2:27:00 PM
Tetrachloroethene	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
1,1,2,2-Tetrachloroethane	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
Toluene	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
Chlorobenzene	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
Ethylbenzene	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
Styrene	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
m,p-Xylene	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM
o-Xylene	< 5.0	5.0		µg/L	1	7/1/2004 2:27:00 PM

Qualifiers:

ND - Not Detected at the Reporting Limit
J - Analyte detected below quanititation limits
B - Analyte detected in the associated Method Blank
* - Value exceeds Maximum Contaminant Level
S - Spike Recovery outside accepted recovery limits
R - RPD outside accepted recovery limits
E - Value above quantitation range

Adirondack Environmental Services, Inc

Date: *02-Jul-04*

CLIENT: Malcolm Pirnie, Inc.
Lab Order: 040701031
Project: WVA-BLDG 40
Lab ID: 040701031-002

Client Sample ID: Trip Blank Lot#091
Collection Date: 7/1/2004
Matrix: WATER

Analyses	Result	PQL	Qual	Units	DF	Date Analyzed
VOLATILE ORGANICS		**SW8260B**				Analyst: **ML**
Chloromethane	< 10	10		µg/L	1	7/2/2004 10:59:00 AM
Bromomethane	< 10	10		µg/L	1	7/2/2004 10:59:00 AM
Vinyl chloride	< 10	10		µg/L	1	7/2/2004 10:59:00 AM
Chloroethane	< 10	10		µg/L	1	7/2/2004 10:59:00 AM
Methylene chloride	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
Acetone	11	10		µg/L	1	7/2/2004 10:59:00 AM
Carbon disulfide	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
1,1-Dichloroethene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
1,1-Dichloroethane	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
trans-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
cis-1,2-Dichloroethene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
Chloroform	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
1,2-Dichloroethane	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
2-Butanone	< 10	10		µg/L	1	7/2/2004 10:59:00 AM
1,1,1-Trichloroethane	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
Carbon tetrachloride	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
Bromodichloromethane	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
1,2-Dichloropropane	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
cis-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
Trichloroethene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
Dibromochloromethane	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
1,1,2-Trichloroethane	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
Benzene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
trans-1,3-Dichloropropene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
Bromoform	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
4-Methyl-2-pentanone	< 10	10		µg/L	1	7/2/2004 10:59:00 AM
2-Hexanone	< 10	10		µg/L	1	7/2/2004 10:59:00 AM
Tetrachloroethene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
1,1,2,2-Tetrachloroethane	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
Toluene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
Chlorobenzene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
Ethylbenzene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
Styrene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
m,p-Xylene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM
o-Xylene	< 5.0	5.0		µg/L	1	7/2/2004 10:59:00 AM

Qualifiers:

ND - Not Detected at the Reporting Limit
J - Analyte detected below quanititation limits
B - Analyte detected in the associated Method Blank
* - Value exceeds Maximum Contaminant Level
S - Spike Recovery outside accepted recovery limits
R - RPD outside accepted recovery limits
E - Value above quantitation range

MW-81 HydroGeophysical Summary

Geophex, Ltd.
605 Mercury Street, Raleigh, NC 27603
Tel: (919) 839-8515 Fax: (919) 839-0096

Job Number: 1282
CLIENT: Malcolm Pirnie, Inc

RECC

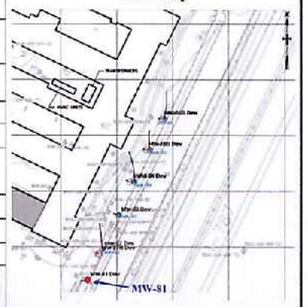
PROJECT: Watervliet Arsenal

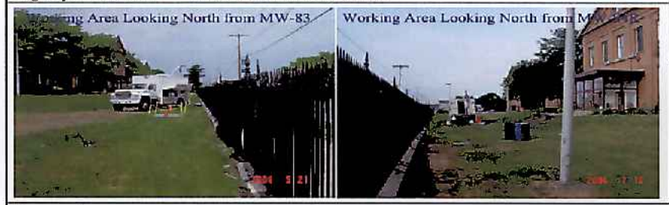
LOCATION: Watervliet, New York

WELL ID: MW-81

Easting:	**Northing:**	**Elevation:**
Drill Date: 10/2003	**Drill Depth:** 150	**Drill Method:** Hammer
Date(s) Logged: 5/19/04 Geophysical Logs 5/26/04 Flow Logging	**File(s):** MW81 Summary Plot.wel	
Casing Stick-Up: -.25-ft	**Casing Diameter:** 6-inch	**Casing Material:** PVC
Bottom of Casing: 20-ft TOC	**Fluid Level:** 10.3-ft bgs	
Log Depth Reference: Logs depth referenced to ground surface.		

Site Map





Comments:

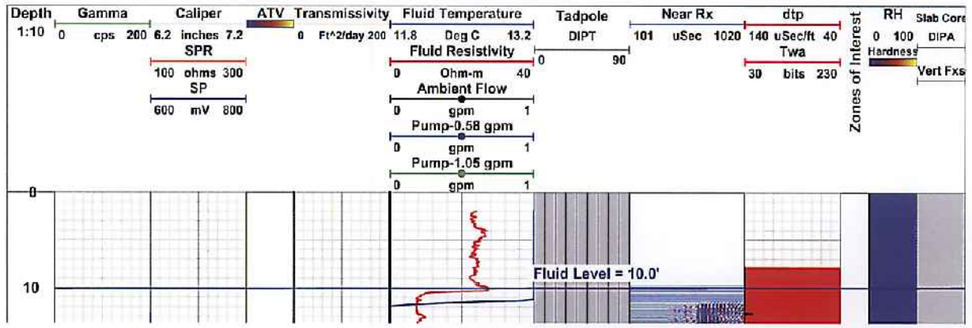
Image Logs corrected for Magnetic Declination of 14.25 deg W and oriented to True North .
Summary Plot contains data from multiple geophysical probes and ambient and pumping flow tests.

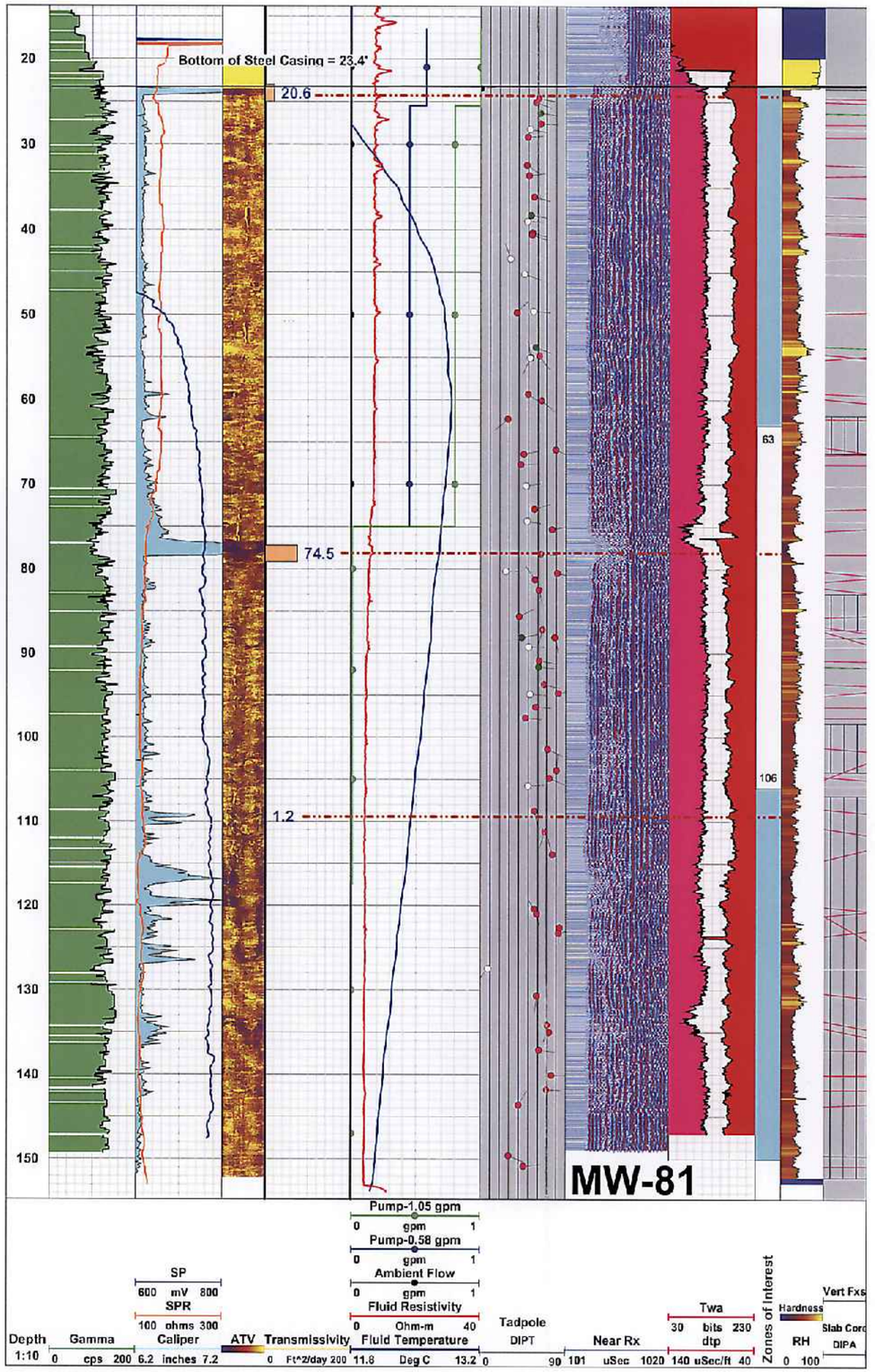
Image Log Feature Legend:

- Thin fracture/joint, discontinuous around circumference
- Indeterminant planar feature - fracture/joint or bedding
- Bedding planar feature
- Wide fracture continuous around circumference
- White in-filled fracture/joint (Calcite?)

Mnemonics:

Gamma	Natural Gamma plotted from 0 to 200 cps & shaded in green
Caliper	3-arm caliper plotted 6.2 to 7.2 inches shaded in light blue
SP	Spontaneous Potential plotted 600 to 800 millivolts in orange
SPR	Single point resistance plotted 100 to 300 ohms in dark blue
ATV	Acoustic televiewer amplitude 2D image - hot yellow colors = high amplitude
Transmissivity	Transmissivity calculated from flow tests - orange bar graph in ft^2/day
Fluid Temperature	Temperature plotted from 11.8 to 13.2 degrees C in blue
Fluid Resistivity	Resistivity of borehole fluid in red from 0 to 40 ohm-meters
Ambient Flow	None-pumping borehole fluid flow plotted from 0 to 1 gpm in black
Pumping 0.58 gpm	Borehole fluid flow while pumping 0.58 gpm plotted 0-1 gpm in blue
Pumping 1.05 gpm	Borehole fluid flow while pumping 1.05 gpm plotted 0-1 gpm in grn
Tadpole	Tadpole plot of image log features corrected to true orientation - see legend
Near Rx	Sonic full waveform from near (3') reciever in micro-seconds
dtp	P or compressional wave transit time (or slowness) in micro-seconds/foot
Twa	Tube or Stonely wave amplidute in bits
Zones of Interest	Three zones of interest and to be monitored during remediation
RH	Relative rock hardness determined from normalized ATV amplitude in %
Hardness	Image log determined from rock hardness log - yellow tones = harder rock
Slab Core	2D projection of image logs on N-S plane thru hole - see legend
Vert Fxs	Near vertical centerline or natural fractures from image logs





MW-82R HydroGeophysical Summary

Geophex, Ltd.
605 Mercury Street, Raleigh, NC 27603
Tel: (919) 839-8515 Fax: (919) 839-0096

Job Number: 1282
CLIENT: Malcolm Pirnie, Inc

RECC

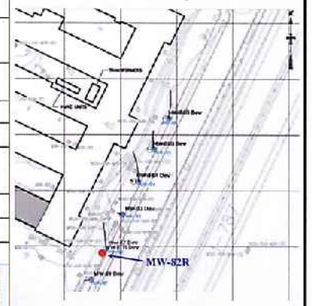
PROJECT: Watervliet Arsenal

LOCATION: Watervliet, New York

WELL ID: MW-82R

Easting:	**Northing:**	**Elevation:**
Drill Date: 6/21/2004	**Drill Depth:** 151	**Drill Method:** Hammer
Date(s) Logged: 7/14/04 Geophysical Logs, 7/15/04 Flow Logging	**File(s):** MW82R Summary Plot.wcl	
Casing Stick-Up: +.33-ft	**Casing Diameter:** 6-inch	**Casing Material:** Steel
Bottom of Casing: 20.07-ft TOC	**Fluid Level:** 10.8-ft bgs	
Log Depth Reference: Logs depth referenced to ground surface.		

Site Map



Comments:

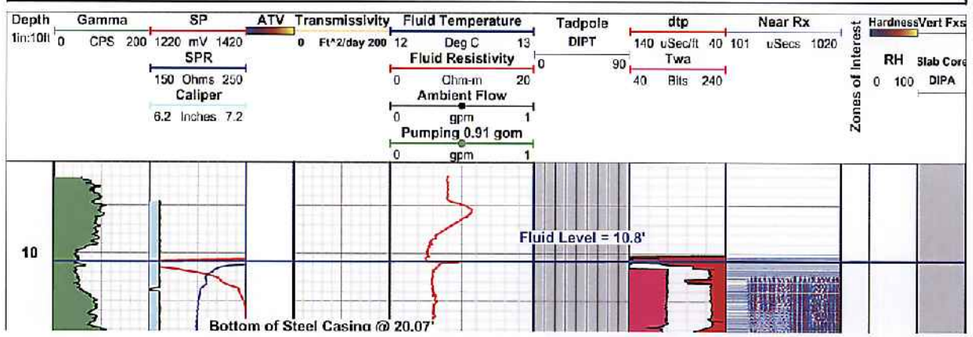
MW-82R is a replacement well located ~9.5' South of MW-82.
Image Logs corrected for Magnetic Declination of 14.25 deg W and oriented to True North .
Summary Plot contains data from multiple geophysical probes and ambient and pumping flow tests.

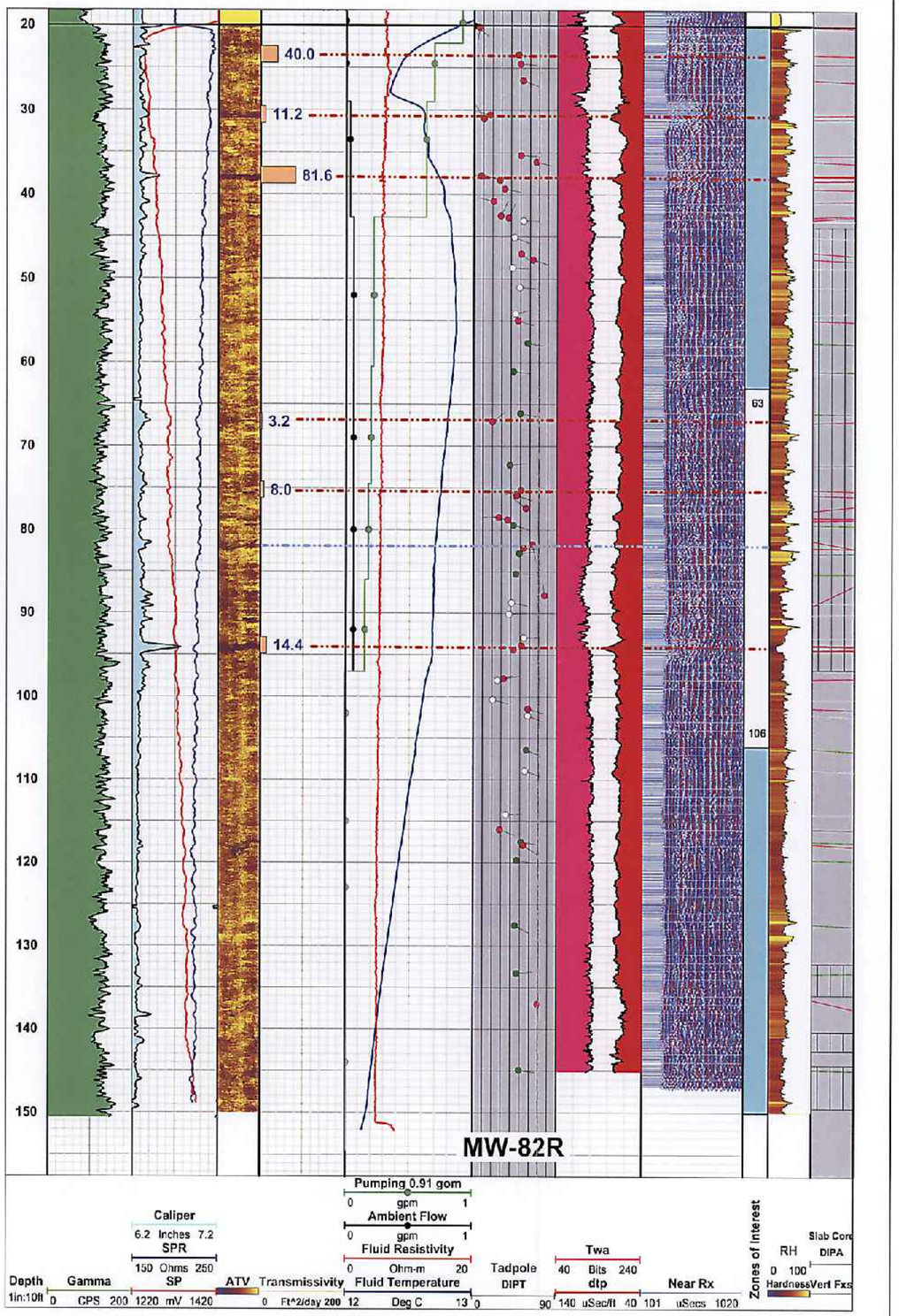
Image Log Feature Legend:

- White in-filled fracture/joint (Calcite?)
- Wide fracture continuous around circumference
- Bedding planar feature
- Indeterminant planar feature - fracture/joint or bedding
- Thin fracture/joint, discontinuous around circumference

Mnemonics:

Gamma	Natural Gamma plotted from 0 to 200 cps & shaded in green
Caliper	3-arm caliper plotted 6.2 to 7.2 inches shaded in light blue
SP	Spontaneous Potential plotted 1220 to 1420 millivolts in orange
SPR	Single point resistance plotted 150 to 250 ohms in dark blue
ATV	Acoustic televiewer amplitude 2D image - hot yellow colors = high amplitude
Transmissivity	Transmissivity calculated from flow tests - orange bar graph in ft^2/day
Fluid Temperature	Temperature plotted from 12.0 to 13.0 degrees C in blue
Fluid Resistivity	Resistivity of borehole fluid in red from 0 to 20 ohm-meters
Ambient Flow	None-pumping borehole fluid flow plotted from 0 to 1 gpm in black
Pumping 0.91 gpm	Borehole fluid flow while pumping 0.91 gpm plotted 0-1 gpm in grn
Tadpole	Tadpole plot of image log fentures corrected to true orientation - see legend
Near Rx	Sonic full waveform from near (3') reciever in micro-seconds
dtp	P or compressional wave transit time (or slowness) in micro-seconds/foot
Twa	Tube or Stonely wave amplitude in bits
Zones of Interest	Three zones of interest and to be monitored during remediation
RH	Relative rock hardness determined from normalized ATV amplitude in %
Hardness	Image log determined from rock hardness log - yellow tones = harder rock
Slab Core	2D projection of image logs on N-S plane thru hole - see legend
Vert Fxs	Near vertical centerline or natural fractures from image logs





MW-83 HydroGeophysical Summary

Geophex, Ltd.
605 Mercury Street, Raleigh, NC 27603
Tel: (919) 839-8515 Fax: (919) 839-0096

Job Number: 1282
CLIENT: Malcolm Pirnie, Inc

RECC

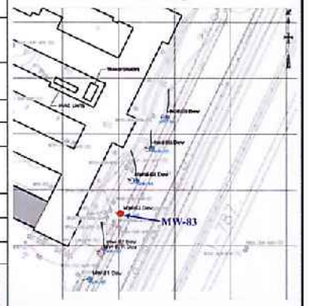
PROJECT: Watervliet Arsenal

LOCATION: Watervliet, New York

WELL ID: MW-83

Easting:	**Northing:**	**Elevation:**
Drill Date: 10/2003	**Drill Depth:** 200	**Drill Method:** Core - reamed w/ rotary
Date(s) Logged: 5/20-21/04 Geophysical Logs, 7/13/04 Flow Logging	**File(s):** MW83 Summary Plot.wcl	
Casing Stick-Up: -.38-ft	**Casing Diameter:** 6-inch	**Casing Material:** PVC
Bottom of Casing: 18.77-ft TOC	**Fluid Level:** 9.77-ft bgs	
Log Depth Reference: Logs depth referenced to ground surface.		

Site Map



Comments:

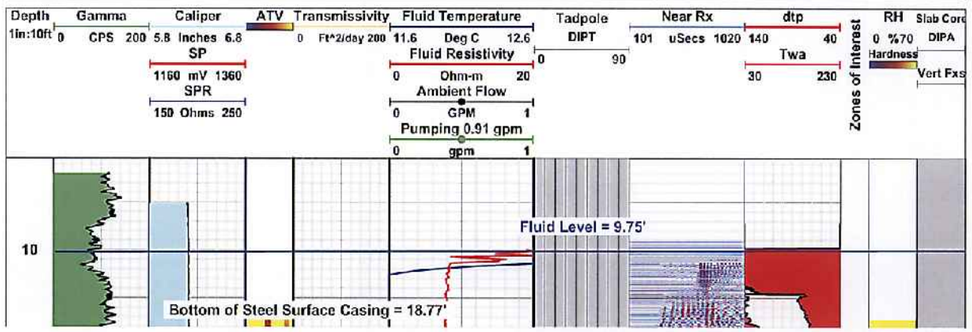
MW-83 was cored and then reamed with 6" rotary bit prior to logging
Image Logs corrected for Magnetic Declination of 14.25 deg W and oriented to True North .
Summary Plot contains data from multiple geophysical probes and ambient and pumping flow tests.

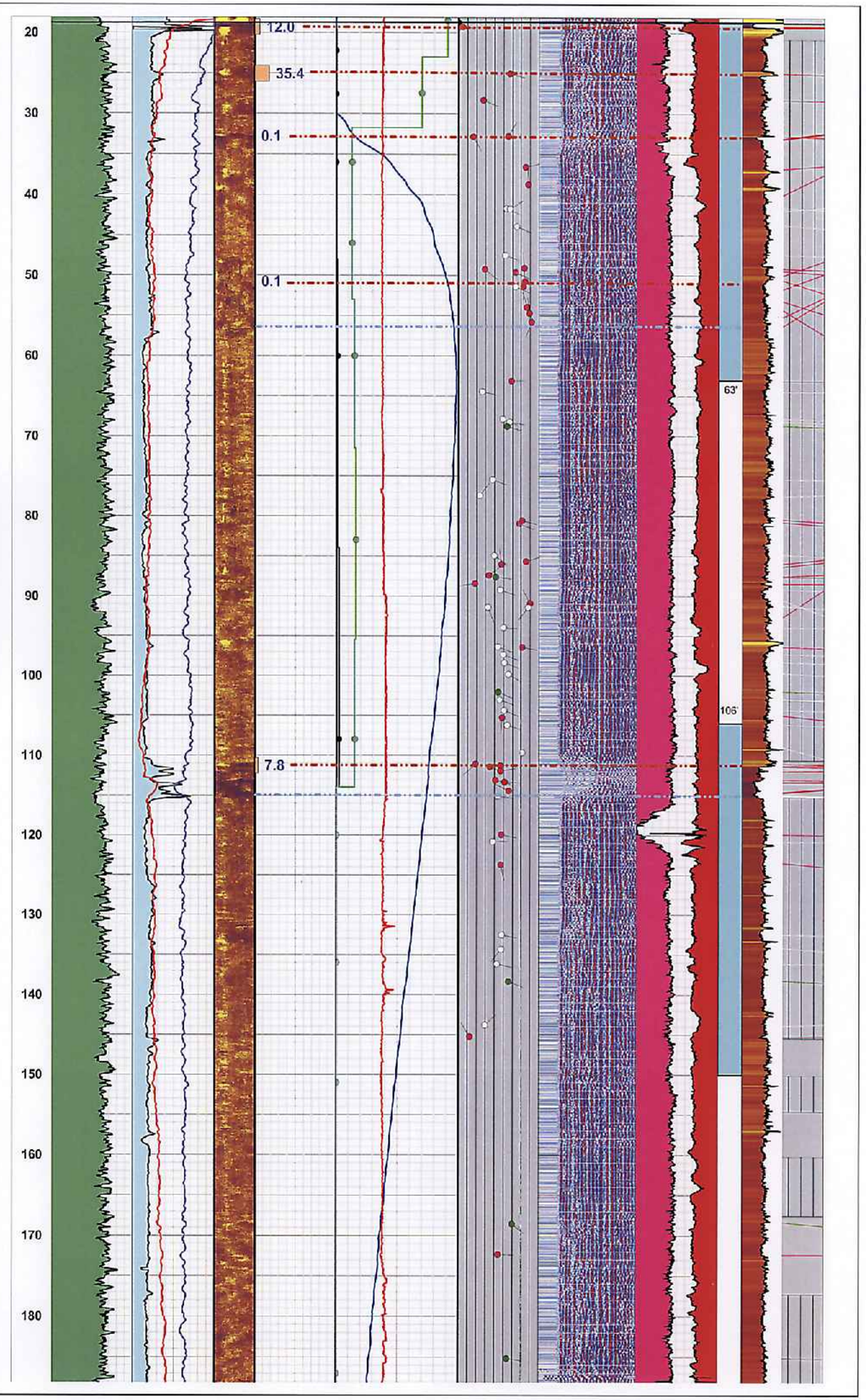
Image Log Feature Legend:

- White in-filled fracture/joint (Calcite?)
- Wide fracture continuous around circumference
- Bedding planar feature
- Indeterminant planar feature - fracture/joint or bedding
- Thin fracture/joint, discontinuous around circumference

Mnemonics:

Gamma	Natural Gamma plotted from 0 to 200 cps & shaded in green
Caliper	3-arm caliper plotted 5.8 to 6.8 inches shaded in light blue
SP	Spontaneous Potential plotted 1160 to 1360 millivolts in orange
SPR	Single point resistance plotted 150 to 250 ohms in dark blue
ATV	Acoustic televiewer amplitude 2D image - hot yellow colors = high amplitude
Transmissivity	Transmissivity calculated from flow tests - orange bar graph in ft^2/day
Fluid Temperature	Temperature plotted from 11.6 to 12.6 degrees C in blue
Fluid Resistivity	Resistivity of borehole fluid in red from 0 to 20 ohm-meters
Ambient Flow	None-pumping borehole fluid flow plotted from 0 to 1 gpm in black
Pumping 0.91 gpm	Borehole fluid flow while pumping 0.91 gpm plotted 0-1 gpm in blue
Tadpole	Tadpole plot of image log features corrected to true orientation - see legend
Near Rx	Sonic full waveform from near (3') receiver in micro-seconds
dtp	P or compressional wave transit time (or slowness) in micro-seconds/foot
Twa	Tube or Stonely wave amplidute in bits
Zones of interest	Three zones of interest and to be monitored during remediation
RH	Relative rock hardness determined from normalized ATV amplitude in %
Hardness	Image log determined from rock hardness log - yellow tones = harder rock
Slab Core	2D projection of image logs on N-S plane thru hole - see legend
Vert Fxs	Near vertical centerline or natural fractures from image logs





MW-84R HydroGeophysical Summary

Geophex, Ltd.
605 Mercury Street, Raleigh, NC 27603
Tel: (919) 839-8515 Fax: (919) 839-0096

Job Number: 1282
CLIENT: Malcolm Pirnie, Inc

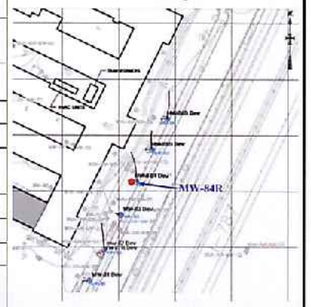
PROJECT: Watervliet Arsenal

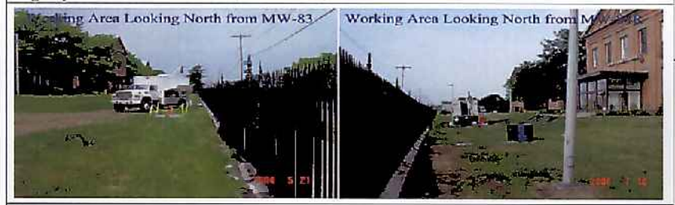
LOCATION: Watervliet, New York

WELL ID: MW-84R

Easting:	**Northing:**	**Elevation:**
Drill Date: 6/29/2004	**Drill Depth:** 151	**Drill Method:** Hammer
Date(s) Logged: 7/12/04 Geophysical Logs, 7/13/04 Flow Logging	**File(s):** MW84R Summary Plot.wel	
Casing Stick-Up: -46-ft	**Casing Diameter:** 6-inch	**Casing Material:** Steel
Bottom of Casing: 22.12-ft TOC	**Fluid Level:** 9.78-ft bgs	
Log Depth Reference: Logs depth referenced to ground surface.		

Site Map





Comments:

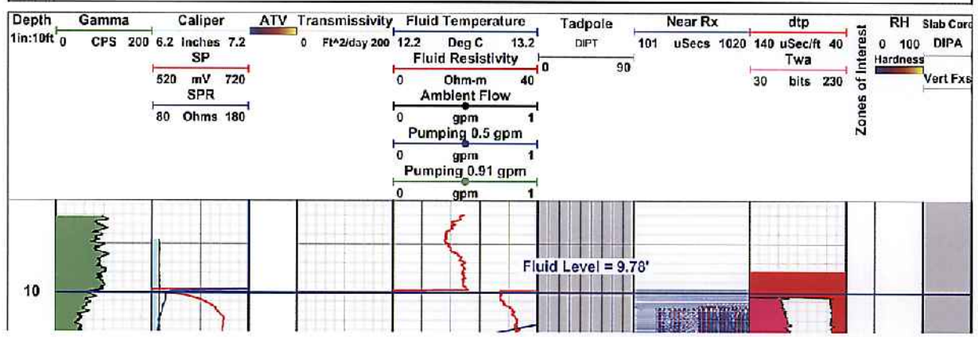
MW-84R is a replacement well located ~8' West of MW-84.
Image Logs corrected for Magnetic Declination of 14.25 deg W and oriented to True North .
Summary Plot contains data from multiple geophysical probes and ambient and pumping flow tests.

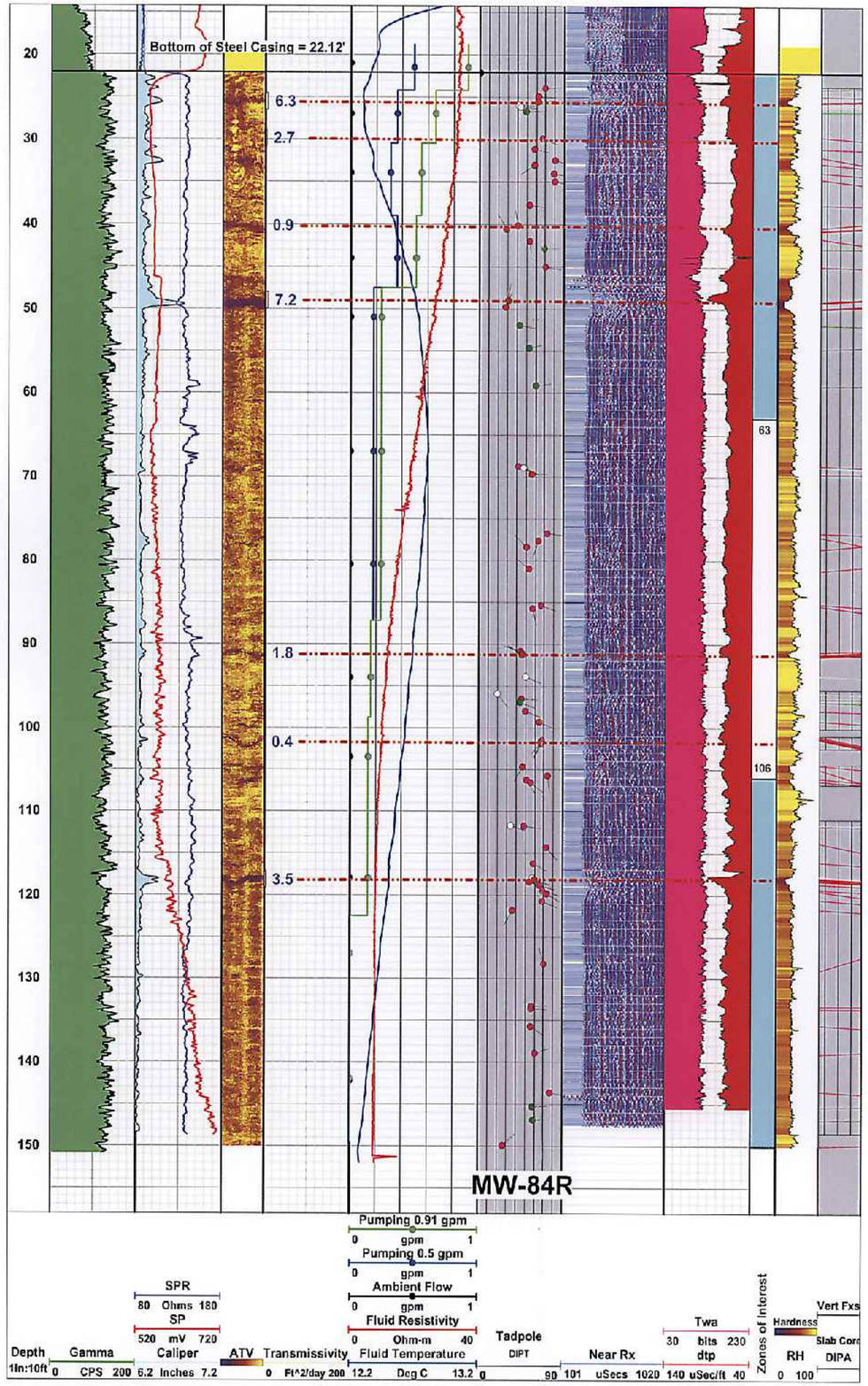
Image Log Feature Legend:

- Thin fracture/joint, discontinuous around circumference
- Indeterminant planar feature - fracture/joint or bedding
- Bedding planar feature
- Wide fracture continuous around circumference
- White in-filled fracture/joint (Calcite?)

Mnemonics:

Gamma	Natural Gamma plotted from 0 to 200 cps & shaded in green
Caliper	3-arm caliper plotted 6.2 to 7.2 inches shaded in light blue
SP	Spontaneous Potential plotted 520 to 720 millivolts in orange
SPR	Single point resistance plotted 80 to 180 ohms in dark blue
ATV	Acoustic televiewer amplitude 2D image - hot yellow colors = high amplitude
Transmissivity	Transmissivity calculated from flow tests - orange bar graph in ft^2/day
Fluid Temperature	Temperature plotted from 12.2 to 13.2 degrees C in blue
Fluid Resistivity	Resistivity of borehole fluid in red from 0 to 20 ohm-meters
Ambient Flow	None-pumping borehole fluid flow plotted from 0 to 1 gpm in black
Pumping 0.50 gpm	Borehole fluid flow while pumping 0.50 gpm plotted 0-1 gpm in gm
Pumping 0.91 gpm#1	Borehole fluid flow while pumping 0.91 gpm plotted 0-1 gpm in gm
Tadpole	Tadpole plot of image log features corrected to true orientation - see legend
Near Rx	Sonic full waveform from near (3') receiver in micro-seconds
dtp	P or compressional wave transit time (or slowness) in micro-seconds/foot
Twa	Tube or Stoneley wave amplitude in bits
Zones of Interest	Three zones of interest and to be monitored during remediation
RH	Relative rock hardness determined from normalized ATV amplitude in %
Hardness	Image log determined from rock hardness log - yellow tones = harder rock
Slab Core	2D projection of image logs on N-S plane thru hole - see legend
Vert Fxs	Near vertical centerline or natural fractures from image logs





MW-85R HydroGeophysical Summary

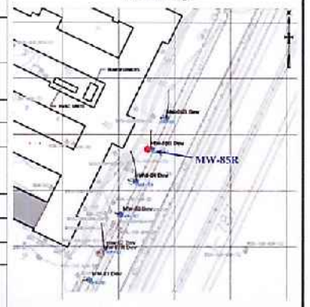
Geophex, Ltd.
605 Mercury Street, Raleigh, NC 27603
Tel: (919) 839-8515 Fax: (919) 839-0096

Job Number: 1282
CLIENT: Malcolm Pirnie, Inc

PROJECT: Watervliet Arsenal
LOCATION: Watervliet, New York
WELL ID: MW-85R

Easting:	**Northing:**	**Elevation:**
Drill Date: 6/21/2004	**Drill Depth:** 150	**Drill Method:** Hammer
Date(s) Logged: 7/09-10/04 Geophysical Logs 7/10/04 Flow Logging	**File(s):** MW85R Summary Plot.wcl	
Casing Stick-Up: -30-ft	**Casing Diameter:** 6-inch	**Casing Material:** Steel
Bottom of Casing: 23.2-ft TOC	**Fluid Level:** 9.84-ft bgs	
Log Depth Reference: Logs depth referenced to ground surface.		

Site Map



Comments:

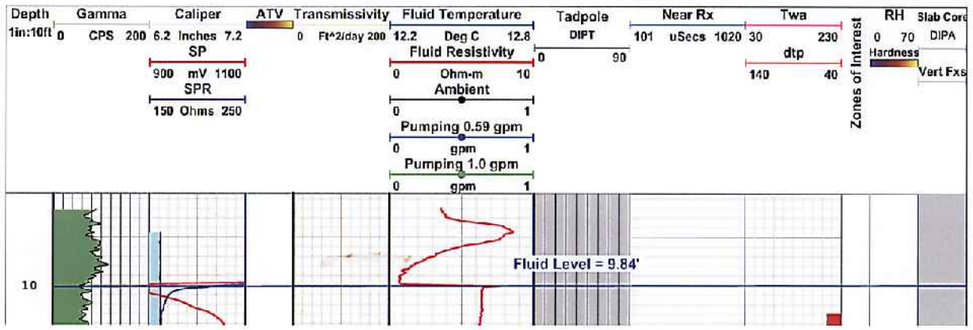
MW-85R is a replacement well located ~7' West of MW-85.
Image Logs corrected for Magnetic Declination of 14.25 deg W and oriented to True North .
Summary Plot contains data from multiple geophysical probes and ambient and pumping flow tests.

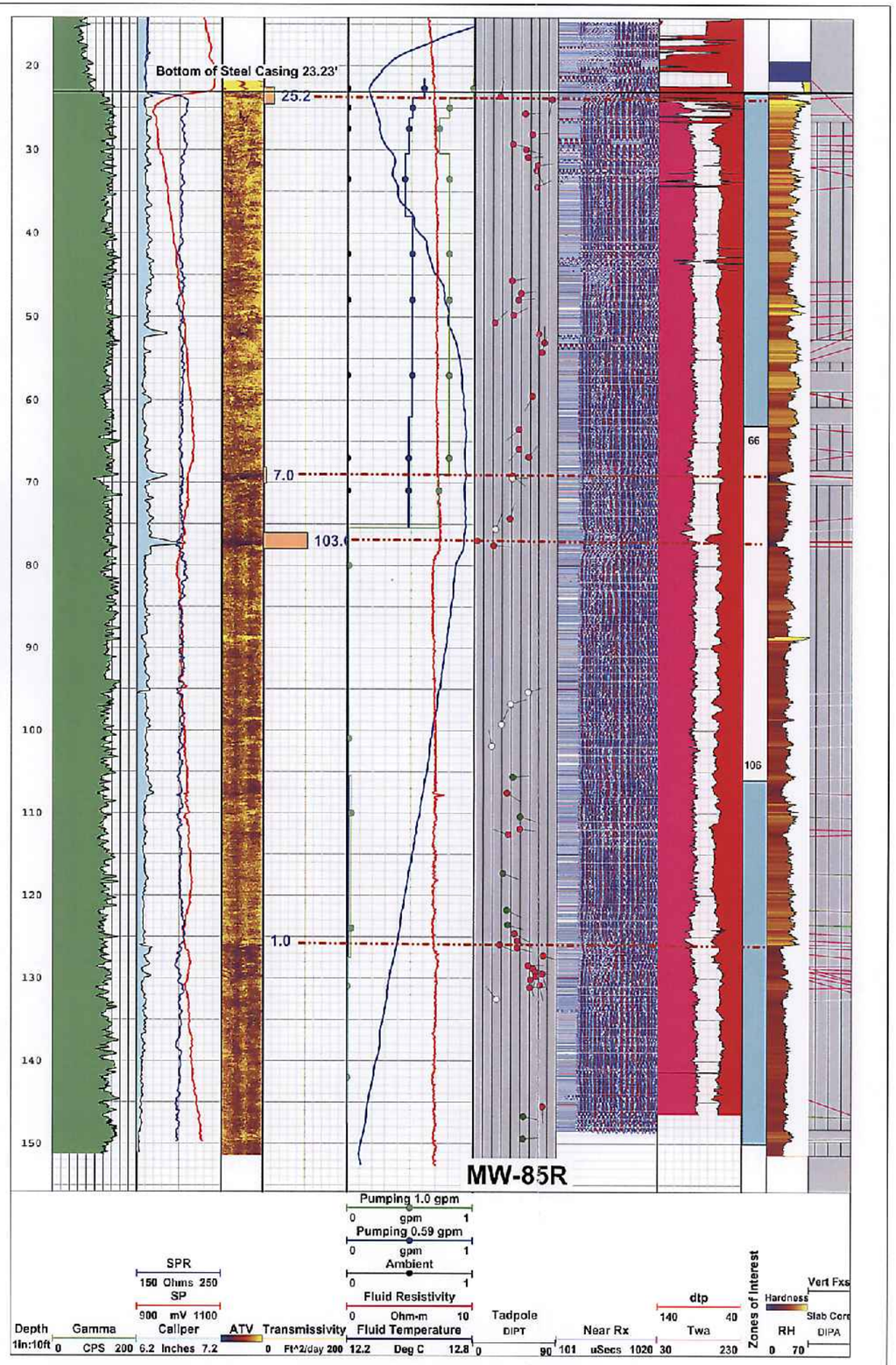
Image Log Feature Legend:

- White in-filled fracture/joint (Calcite?)
- Wide fracture continuous around circumference
- Bedding planar feature
- Indeterminant planar feature - fracture/joint or bedding
- Thin fracture/joint, discontinuous around circumference

Mnemonics:

Gamma	Natural Gamma plotted from 0 to 200 cps & shaded in green
Caliper	3-arm caliper plotted 6.2 to 7.2 inches shaded in light blue
SP	Spontaneous Potential plotted 900 to 1100 millivolts in orange
SPR	Single point resistance plotted 150 to 250 ohms in dark blue
ATV	Acoustic televiewer amplitude 2D image - hot yellow colors = high amplitude
Transmissivity	Transmissivity calculated from flow tests - orange bar graph in ft^2/day
Fluid Temperature	Temperature plotted from 12.2 to 12.8 degrees C in blue
Fluid Resistivity	Resistivity of borehole fluid in red from 0 to 10 ohm-meters
Ambient Flow	None-pumping borehole fluid flow plotted from 0 to 1 gpm in black
Pumping 0.59 gpm	Borehole fluid flow while pumping 0.59 gpm plotted 0-1 gpm in blue
Pumping 1.0 gpm	Borehole fluid flow while pumping 1.0 gpm plotted 0-1 gpm in grn
Tadpole	Tadpole plot of image log features corrected to true orientation - see legend
Near Rx	Sonic full waveform from near (3') reciever in micro-seconds
dtp	P or compressional wave transit time (or slowness) in micro-seconds/foot
Twa	Tube or Stonely wave amplidute in bits
Zones of interest	Three zones of interest and to be monitored during remediation
RH	Relative rock hardness determined from normalized ATV amplitude in %
Hardness	Image log determined from rock hardness log - yellow tones = harder rock
Slab Core	2D projection of image logs on N-S plane thru hole - see legend
Vert Fxs	Near vertical centerline or natural fractures from image logs





MW-86R HydroGeophysical Summary

Geophex, Ltd.
605 Mercury Street, Raleigh, NC 27603
Tel: (919) 839-8515 Fax: (919) 839-0096

Job Number: 1282
CLIENT: Malcolm Pirnie, Inc

RECC

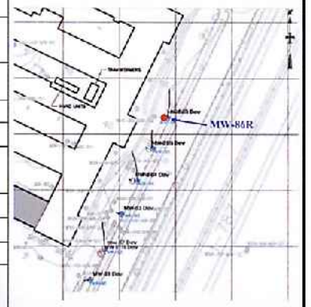
PROJECT: Watervliet Arsenal

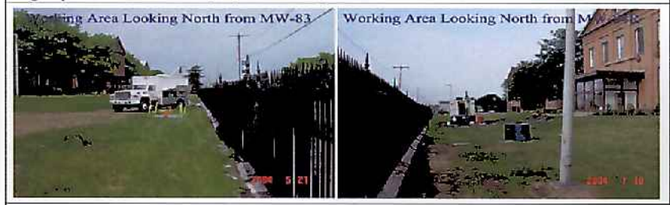
LOCATION: Watervliet, New York

WELL ID: MW-86R

Easting:	Northing:	Elevation:
Drill Date: 6/16/2004	**Drill Depth:** 151	**Drill Method:** Hammer
Date(s) Logged: 7/08/04 Geophysical Logs 7/09/04 Flow Logging	**File(s):** MW86R Summary Plot.wcl	
Casing Stick-Up: -.25-ft	**Casing Diameter:** 6-inch	**Casing Material:** Steel
Bottom of Casing: 23.74-ft TOC	**Fluid Level:** 10.17-ft bgs	
Log Depth Reference: Logs depth referenced to ground surface.		

Site Map





Comments:

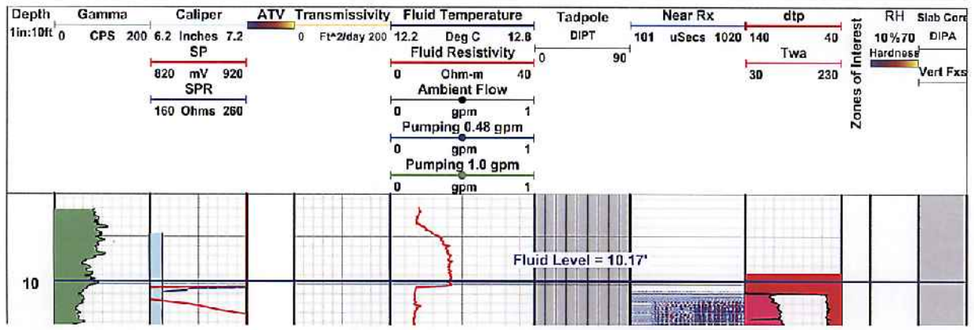
MW-86R is a replacement well located ~6' West of MW-86.
Image Logs corrected for Magnetic Declination of 14.25 deg W and oriented to True North.
Summary Plot contains data from multiple geophysical probes and ambient and pumping flow tests.

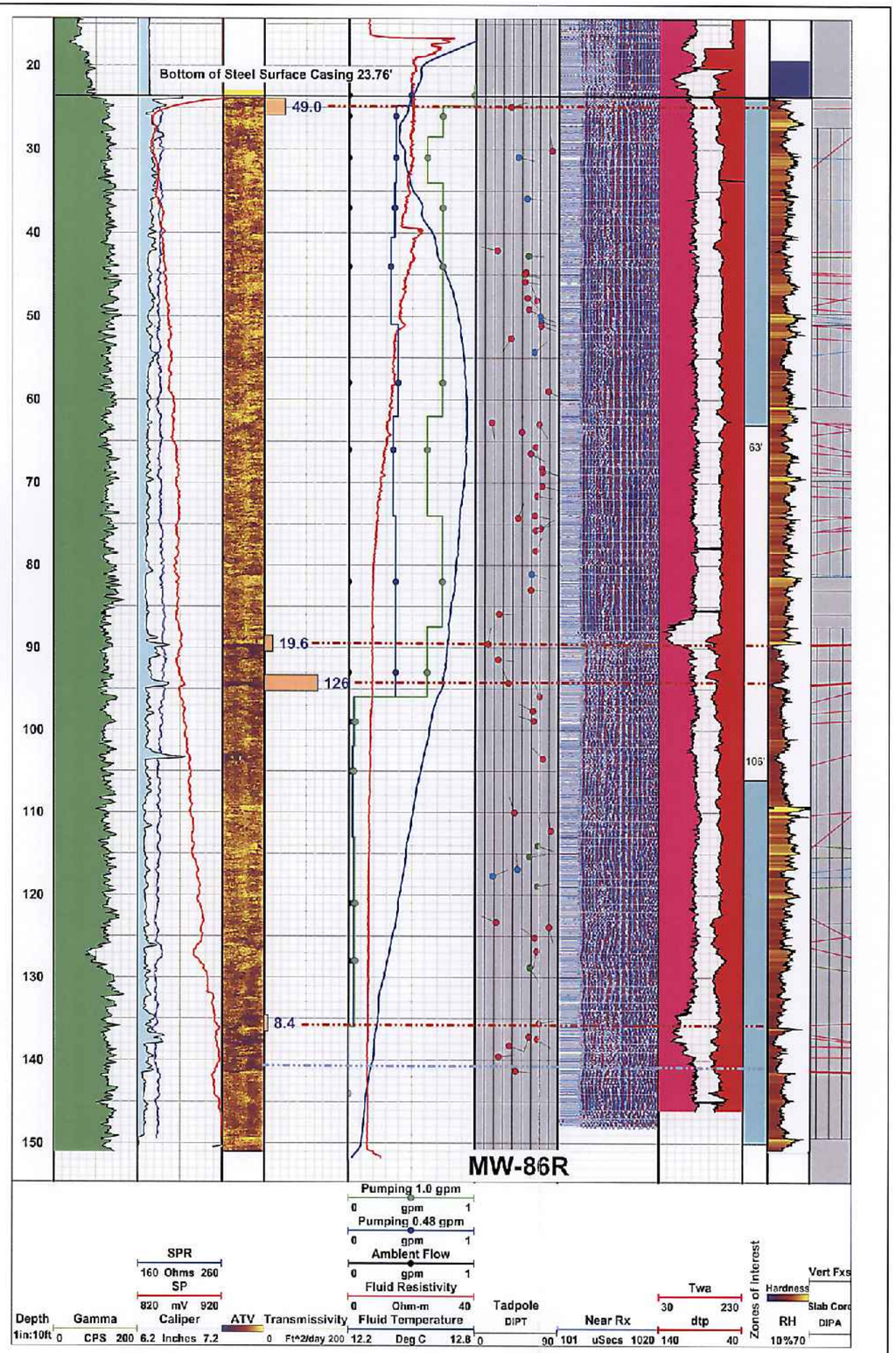
Image Log Feature Legend:

- Thin fracture/joint, discontinuous around circumference
- Indeterminant planar feature - fracture/joint or bedding
- Bedding planar feature
- Wide fracture continuous around circumference
- White in-filled fracture/joint (Calcite?)

Mnemonics:

Gamma	Natural Gamma plotted from 0 to 200 cps & shaded in green
Caliper	3-arm caliper plotted 6.2 to 7.2 inches shaded in light blue
SP	Spontaneous Potential plotted 820 to 920 millivolts in orange
SPR	Single point resistance plotted 160 to 260 ohms in dark blue
ATV	Acoustic televiewer amplitude 2D image - hot yellow colors = high amplitude
Transmissivity	Transmissivity calculated from flow tests - orange bar graph in ft^2/day
Fluid Temperature	Temperature plotted from 12.2 to 12.8 degrees C in blue
Fluid Resistivity	Resistivity of borehole fluid in red from 0 to 40 ohm-meters
Ambient Flow	None-pumping borehole fluid flow plotted from 0 to 1 gpm in black
Pumping 0.48 gpm	Borehole fluid flow while pumping 0.48 gpm plotted 0-1 gpm in blue
Pumping 1.0 gpm	Borehole fluid flow while pumping 1.0 gpm plotted 0-1 gpm in gm
Tadpole	Tadpole plot of image log features corrected to true orientation - see legend
Near Rx	Sonic full waveform from near (3') reciever in micro-seconds
dtp	P or compressional wave transit time (or slowness) in micro-seconds/foot
Twa	Tube or Stonely wave amplidute in bits
Zones of Interest	Three zones of interest and to be monitored during remediation
RH	Relative rock hardness determined from normalized ATV amplitude in %
Hardness	Image log determined from rock hardness log - yellow tones = harder rock
Slab Core	2D projection of image logs on N-S plane thru hole - see legend
Vert Fxs	Near vertical centerline or natural fractures from image logs





Severn Trent Laboratories - Monroe

SOP for GC/MS Volatiles SW846 8260B

MSS02803.CT
Revision 3
Date Effective: 11/05/1999

This documentation has been prepared by Severn Trent Laboratories (STL) solely for STL's own use and the use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to Severn Trent Laboratories upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF SEVERN TRENT LABORATORIES IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY SEVERN TRENT LABORATORIES IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING

This is a controlled Document if Stamped in Red:

CONTROLLED COPY # 1

Issued to: Marie Vida

1.0 APPROVALS

The signatures of the following individuals indicates that this SOP is complete and meets the requirements specified in the SOP for SOPs.

Laboratory Director

Quality Assurance Manager

GG/MS Manager

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to outline the techniques for determining the presence and concentration of various volatile organic target and non-target compounds in multi-media, multi-concentration samples. The 8260B compounds for this method are listed in Table 1.0. Table 2.0 lists the expanded Appendix IX target compounds which are applicable to this method. The extraction method used in this procedure is purge and trap which is coupled with a gas chromatograph/mass spectrometer analysis.

2.2 It is the policy of STL and of the GC/MS Group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the STL Policy Statement on Business Ethics and Conduct.

2.3 The document control number for this SOP is MSS02803.CT.

3.0 Terms and Definitions

3.1 There are many definitions used within the laboratory, which may be generic to all Laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used within the laboratory, reference the SOP of Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This method employs the technique of purge and trap, coupled with a gas chromatograph/mass spectrometer analysis. An aliquot of sample, usually 5 ml or 25 ml of water, 5 g of soil for low level soil method, and 5 g of soil collected with methanol and extracted for medium level soil method, is purged in a gas tight chamber with UHP grade helium to remove the volatile compounds. The vapor is swept through a sorbent column where the volatiles are trapped. Next the sorbent trap is heated and back flushed, thereby

desorbing the volatiles onto the analytical column within the gas chromatograph. The fused silica capillary column is then temperature programmed to separate the volatiles prior to detection by the mass spectrometer.

4.2 This SOP is based on USEPA SW846 5030B/5035/8260B Methods.

4.3 The following deviations from the method are noted:

. The selected internal standards for this 8260B Method are:

- Bromochloromethane
- 1,4-Diflourobenzene
- Chlorobenzene-d_5

. The selected surrogates for this 8260B Method are:

- 1,2-Dichloroethane-d_4
- Toluene-d_8
- Bromofluorobenzene

. The routine 8260B target list is the TCL list per Table 1.0.

. Method 8260B can be either 5 ml or 25 ml purge per methodology. This must be specified by the client prior to sample analysis. In many cases the detection limits requested can be achieved by 5 ml sample volume.

5.0 INTERFERENCES

5.1 Method interferences may be caused by contaminants in solvents, reagents, out-gassing from equipment plumbing, and laboratory solvent vapors. This can lead to discrete artifacts and/or elevated baseline in the gas chromatograph. All these materials must be demonstrated to be free from interferences by the running of laboratory reagent blanks.

5.2 Interferences may also be caused by the diffusion of volatiles through the septum seal during storage and handling. A holding blank prepared from reagent water is stored with the samples and analyzed to serve as a check. Holding blanks are prepared weekly for each volatile sample storage unit. The results for the holding blank are reported by GC/MS and submitted to the QA/QC Manager to be filed for future reference.

5.3 Only Baxter Purge and Trap grade methanol shall be used for standards and sample dilutions.

5.4 Only reagent water free from Volatile organics can be used in the volatiles laboratory for sample dilutions, reagent blanks, and aqueous standards.

6.0 SAFETY

6.1 Analysts shall treat all samples as if they are hazardous and take all appropriate safety precautions. Analysts shall wear, if needed:

- lab coats
- safety glasses with side shields and
- chemical resistant gloves

when handling samples or preparing standards.

6.2 Solvents and all standards shall be used in the fume hoods to minimize environmental exposure to solvent vapors.

6.3 Material Safety Data Sheets for all chemicals used in the operation are present in the laboratory for immediate access.

7.0 SAMPLE PRESERVATION AND STORAGE

7.1 All samples for volatile analysis must be protected from light and refrigerated at 4°C from the time of receipt until analysis.

7.2 All HCL preserved samples for volatile analysis shall be analyzed within 14 days of sample collection, or seven days unpreserved.(the laboratory must be notified if samples are unpreserved to ensure unpreserved samples can be run within holding time) NYSDEC 8260B samples must be analyzed within ten days of receipt.

7.3 Refer to the sample control processing, sample removal, and log-in SOP's in section 15.0.

7.4 Low level soil analysis will be preserved with reagent grade Sodium bisulfate and reagent water. Client must specify at time of bottle order if no preservative is required.

7.5 Methanol preserved vials will be sent out for projects requesting the high concentration soil analysis.

8.0 APPARATUS AND MATERIALS

8.1 Purge and trap concentrator - Tekmar Model 2000 or 3000

8.2 Purge and trap autosampler - Tekmar Model 2016 or Archon 51 position autosampler

8.3 Volatile Trap - Tekmar Number 2 trap (25 cm tenax-GC, 60/80 mesh and 8 cm of silica gel, 35/60 mesh, grade 15, or equivalent); at least 25 cm long with outside diameter of at least 0.105 or equivalent trap such as the VOACARB 3000.

8.5 GC/MS/DS System

8.4.1 Hewlett-Packard Model 5971 or 5970 GC/MS with jet separator, or direct capillary interface, capable of scanning from 35 to 300 amu every two seconds or less, utilizing 70 volts (nominal) electron energy in the EI ionization mode, and producing a mass spectrum which meets all the instrument performance criteria when 50 ng of BFB is injected through the GC inlet. Refer to Sections 10.3.1 for the performance criteria and section 11.1 for the instrumental conditions.

8.4.2 Direct interface from purge and trap transfer line to GC column

8.4.3 GC Column - 75 m x 0.53 mm ID x 3.0 um film thickness Supelco 624 fused silica widebore capillary column, or equivalent.

8.4.4 Chemstation software capable of continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any data file for ions of a specified mass and plotting such ion abundances versus time or scan number (EICP). Software also allows integrating the abundance in any EICP between specified time limits. Also, software allows for the comparison of sample non-target spectrum against reference library spectra. The most recent release of the NIST/EPA/MSDC mass spectral library shall be used as the reference library. The data system flags all manual edits with "M" qualifier.

8.4.5 Chemstation network tape backup system.

8.4.6 Syringes – Gas-tight microsyringes 25 ul and larger, 0.006 inch ID needle; 5 ml gas-tight syringes with shut off valve, all syringes used shall be gas-tight

8.4.7 Balance – top loading balance capable of weighing +/-0.1 g, and an analytical balance capable of weighing +/-0.0001 grams. This balance must be checked for calibration once, prior to use with NIST weights. The range will be from 1gram to 100grams to bracket the working range.

8.4.8 Methanol – Purge and Trap Grade

8.4.9 Fritted sparger or culture tube

8.4.10 Syringe Valve – two way, with Luer ends (three each), if applicable to the purging device

8.4.11 Glassware

- Bottle – 15 ml, screw cap, with Teflon cap liner
- Volumetric flasks – class A with ground glass stoppers
- Vials – 2 ml for GC autosampler

8.4.12 A heater or heated water bath capable of maintaining the purge device at 40°C +/- 1°C attached to a stir plate or sonicator that will agitate the low level soil sample, but not for waters or medium level soils

8.4.13 pH paper – narrow range (0-6pH units)- All aqueous samples will have a pH taken and recorded in the injection logbook. Any pH readings above 2 will be addressed in a corrective action, if samples are analyzed more than seven days from collection.

8.4.14 Magnetic stir bars

8.4.15 Polyethylene glycol (PEG)

9.0 REAGENTS AND STANDARD PREPARATION

Refer to the volatiles standards preparation SOP, referenced in section 15.0, for details of standard preparation.

9.1 Reagent Water - Laboratory certified water, free from contaminants.

9.2 Dilution Methanol - Baxter Purge and Trap Grade or equivalent.

9.3 Stock Standards - certified standards purchased from commercial sources containing ampulated mixes of target compounds, matrix spike compounds, surrogates, and internal standards are used by the laboratory as stock standards. New ampules are opened every two months, or sooner, if the standard has degraded or evaporated

9.4 Working Standards

9.4.1 Instrument performance check solution - 4-Bromofluorobenzene (BFB) 25 ng/ul solution of BFB in methanol is prepared every six months, or sooner if the solution has degraded or evaporated. Add 2 ul of this solution into 5ml of reagent water for a 50ng concentration.

9.4.2 Calibration Standard Solution

A 25 ng/ul working calibration standard containing all the volatile target compounds in methanol is prepared weekly, or sooner, if the solution has degraded or evaporated.

9.4.3 Internal Standard (IS) Spiking Solution

A 50 ng/uL IS spiking solution containing bromochloromethane, chlorobenzene-d_5, and 1,4-difluorobenzene in methanol is prepared weekly, or sooner, if the solution has degraded or evaporated. Add 5 uL of this solution utilizing a 25 uL gas-tight syringe, to 5 ml sample, standard, or blank for a concentration of 50 ug/L.

9.4.4 System Monitoring Compound (SMC) Spiking Solution

A 50 ng/uL SMC spiking solution containing 1,2-dichloroethane-d_4, toluene-d_8, and 4-bromofluorobenzene in methanol is prepared weekly, or sooner, if the solution has degraded or evaporated. Add 5 uL of this solution utilizing a 25 uL gas-tight syringe, to 5 ml sample, standard, or blank for a concentration of 50 ug/L.

9.4.5 Matrix Spiking (MS) Solution

A 50 ng/uL MS solution containing 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene in methanol is prepared weekly, or sooner, if the solution has degraded or evaporated.

9.4.6 FMS Spiking Solution (LCS)

A 50 ng/uL FMS solution containing all TCL compounds is prepared weekly from a source independent of the calibration standard in methanol. This solution is run every time a matrix spike, matrix spike duplicate is analyzed.

10.0 CALIBRATION

10.1 Calibration Standards

Five aqueous initial calibration standards containing all the volatile target compounds and SMC's are prepared at the 5, 20, 50, 100, and 200 ug/L levels. These standards are prepared from working standards in 9.0 (25 ml purge at 1, 4, 10, 20, 40 ug/L levels).

The methanol purged in the aqueous calibration standards must not exceed 1% by volume.

10.2 The working calibration of this method is defined by the initial calibration curve, 5 ug/L

to 200 ug/L. All samples with target compounds exceeding 200 ug/L for a 5 ml purge or 40 ug/l for a 25 ml purge must be diluted to within the upper half of the calibration range. If an automatic liquid sampler (ALS) position is contaminated by a compound, that particular ALS position will be "capped" with a red plastic cap and not used until deemed clean by the analysis of a system blank run in that position. No compounds can be over the Practical Quantitation Limit (PQL) for that particular compound.

10.3 Calibration curve preparation

The calibration curve is prepared by adding the following amounts of working calibration standard and SMC are added to 5 ml, or 25 ml of reagent water, achieving the indicated concentrations:

5 ml Purge		25 ml Purge	
Conc. Level	Volume to Add	Conc. Level	Volume to Add
5 ug/L	1 uL	1 ug/L	1 uL
20 ug/L	4 uL	2 ug/L	2 uL
50 ug/L	10 uL	5 ug/L	5 uL
100 ug/L	20 uL	10 ug/L	10 uL
200 ug/L	40 uL	25 ug/L	25 uL

The IS spiking solution is added at 10 ul utilizing a 25 uL gas-tight syringe, to all calibration standards for a final concentration of 50 ug/L.

An initial calibration must be analyzed on each GC/MS system upon column installation, whenever corrective action is taken which may affect the initial calibration criteria, or if the continuing calibration criteria can not be met.

Separate initial and continuing calibration must be analyzed for water samples, and low level soil samples (unheated versus heated/agitated purge). Extracts of medium level soil samples may be analyzed using the calibrations for water.

Quantitation is based on the average response factor from the initial calibration.

10.4 Acceptance criteria for the Initial Calibration

The initial calibration criteria must meet the following:

- Calibration Check Compounds (CCC) must have RF's whose percent relative standard deviations are less than 30 percent.

System Performance Check Compounds (SPCC) Average RF must be equal to or greater than 0.100 except for chlorobenzene and 1,1,2,2 tetrachloroethane must be 0.30 or higher.

10.4.1 The percent relative standard deviation (%RSD) should be less than 15 % for each compound of interest.

1. If the %RSD is less than 15 % then the average RF from the initial calibration curve is used for quantitation of the compound.

2. If the **mean** %RSD is below 15% for **all** compound in the initial calibration, then average RF can be used, if not, then linear regression is used for quantitation for those compounds above 15%. The minimum correlation coefficient for any compound using linear regression must also be 0.99. A copy of the linear regression plot from the calibration curve shall be included in the raw data.
3. **If the samples are ACOE samples then the initial calibration must meet the following criteria: All compounds less than or equal to 15% RSD with the exception of the poor performing compounds listed below are allowed to be up to 40%RSD**
   Poor performers: Chloromethane, Bromomethane, Vinyl Chloride, Chloroethane, Acetone, Acrolein, Acrylonitrile, 1,4-Dioxane, Trichlorofluoromethane, 2-Butanone, Vinyl Acetate, 2-Chloroethyl(vinyl)ether, 4-Methyl-2-Pentanone, Carbon Disulfide, 2-Nitropropane, Dichlorodifluoromethane, 1,2-Dibromo-3-Chloropropane

   If a compound has a % RSD outside of the criteria (either 15% or 40%) and is a target compound for a particular project, then the problem will be corrected and the system recalibrated.

An independent source spike at 50ug/L is analyzed after the initial calibration, to verify all compounds. The acceptance windows for guidance shall be 75- 125% Recovery. Any compound outside this range shall be brought to the attention of the group leader for monitoring. A copy of the spike shall be given to the QC officer for control charting.

10.5 Continuing calibration preparation

The continuing calibration standard is the 50 ug/L standard from the initial calibration or is a separate standard prepared as above at 50 ug/L.

The continuing calibration standard must be analyzed every 12 hours to verify that the initial calibration is still valid.

10.6 Continuing Calibration Acceptance criteria

- Calibration Check Compounds (CCC) - percent RSD 20 percent maximum
- System Performance Check Compounds (SPCC) - 0.1 minimum RF (0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane)

All other compounds shall be reviewed for accuracy.

ACOE projects must have all compound in the continuing calibration under 20%Difference. Poor performing compounds are allowed to be up to 40%D as specified in the initial calibration criteria section 9.4.1.3.

11.0 QUALITY CONTROL

11.1 Method detection limit determination is required by this method.

11.2 Quantitation limits for this method are defined by the method detection limit, however practical quantitation limits have been set by the laboratory (see Table 1.0).

11.3 Daily Performance Tests

11.3.1 Prior to initiating any data collection activities it is necessary to establish that a given GC/MS system meets the instrument performance criteria. This is accomplished through the analysis of 50 ng of p-bromofluorobenzene (BFB).

11.3.1.1 BFB must be analyzed at the start of every 12 hour sequence. 50 ng of BFB may be directly injected onto the GC column or purged in 5.0 ml of reagent water. BFB may not be analyzed simultaneously with a calibration standard.

11.3.1.2 The key ions produced during the analysis of BFB and their respective ion abundance criteria are given in Table 3.0. This criteria must be met before any calibration standards, blanks, or samples may be analyzed.

11.3.1.3 Use the tuner program to verify that the BFB spectrum is within criteria. If it is not within criteria, the analyst may use enhancing or other acceptable practices to put BFB within criteria.

11.3.1.4 If the criteria is not met, the BFB must be reanalyzed. Repeated failure shall require the instrument to be manually tuned. After manual tuning, the BFB must be re-injected and the abundance criteria must be met before proceeding.

11.3.2 After the instrument performance criteria is met, the initial calibration curve must be verified through the analysis of a continuing calibration at 50 ug/L. The continuing

calibration criteria must be met before any method blank or sample analyses may proceed.

11.3.3 A method blank consisting of 5ml of reagent water spiked with 50 ug/L of IS and SMC's must be analyzed every 12 hours after calibration criteria has been met. An acceptable method blank must meet the following criteria:

PQL's can vary on each project. The PQL's must be reviewed prior to sample analysis.
Less than or equal to **three times** the PQL for method blanks for the target compounds methylene chloride and acetone.

. Less than or **one half** the PQL for all each of the other target compounds

Sample analysis may not proceed until the above method blank criteria has been met.

All volatile analyses associated with a method blank that does not meet the above requirements must be repurged, reanalyzed, and reported.

11.4 Matrix Spike, Matrix Spike Duplicates and Matrix Spike Blanks

11.4.1 An MS/MSD must be analyzed for each group of samples of a similar matrix within each case, 20 samples, group of samples of a similar concentration level (soils only), or each 7 calendar day period; whichever is more frequent. MSB's are required for NYSDEC protocols.

11.4.2 The limits for matrix spike compound recovery and relative percent difference (RPD) are given in Table 4.0. These limits are only advisory; therefore, no further action is required if the criteria limits are not achieved. However, frequent failures shall be investigated for possible laboratory generated error.

11.5 QC Check Samples are applicable to this method. With every MS/MSD, a 20 ug/L QC check sample will be run. This QCS is a full matrix spike prepared from an independent stock solution for comparison against the calibration check.

11.6 System Monitoring Compounds

11.6.1 SMC's are added to each sample, blank, standard, QCS and MS/MSD/MSB, prior to purging or extracting at 50 ug/L for waters, 50 ug/Kg for low level soils, and 5000 ug/Kg for medium level soils.

11.6.2 SMC recoveries must be within the QC limits given in Table 5.0. If the recovery for any one SMC is not within limits, the following are required:

. Check all calculations for accuracy, spiking solutions, and internal standards

- Reanalyze the sample if none of the above steps reveal a problem

- If an undiluted analysis with acceptable SMC recoveries is being submitted, do not reanalyze diluted samples if the SMC recoveries are outside the limits

- Never reanalyze the MS or MSD, even if the SMC recoveries are outside the limits

- If the sample associated with the MS/MSD does not meet specifications, it should be reanalyzed only if the MS/MSD SMC recoveries are within the limits. Document in the narrative the similarity in the SMC recoveries between the sample and associated MS/MSD.

If the reanalysis of the sample solves the problem, then only submit the second analysis. If the reanalysis does not solve the problem, then submit the data from both analyses.

11.6.3 If the recovery of any one SMC in a method blank is outside limits, then the method blank and all associated samples must be reanalyzed.

11.7 Internal Standards

11.8.1 IS's are added to each sample, blank, standard, and MS/MSD/MSB, at 50 ug/L at the time of purging.

11.8.2 The retention times (RT) and extracted ion current profile (EICP) of each IS must be evaluated for all standards immediately after the data acquisition. The IS EICP areas must be monitored and evaluated for each sample, blank, MS, MSD. If the IS EICP changes by more than a factor of 2 (-50% to +100%) from the latest (12 hour) calibration standard, the GC/MS system must be inspected for malfunctions, and corrections made as required. If the RT for any IS changes by more than 30 seconds from the latest (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. For samples analyzed within the same 12 hour time period as the initial calibration standards, compare the IS responses and RT's against the 50 ug/L calibration standard. When corrections are made, reanalysis of the samples analyzed while the system was malfunctioning is necessary.

If IS criteria is not within limits, the following are required:

- Check all calculations for accuracy, spiking solutions, and internal standards

- Reanalyze the sample if none of the above steps reveal a problem

- If the sample associated with the MS/MSD does not meet specifications, it should

be reanalyzed only if the MS/MSD IS criteria is within the limits

. If the reanalysis of the sample solves the problem, then only submit the second analysis. If the reanalysis does not solve the problem, then submit the data from both analyses.

11.8 Quality Control Check Points

11.8.1 Analysis quality control approval report

Specific quality control check points have been established for the analysis of samples which are monitored through a Quality Control Approval Report (QCAR). The specific check points must be initialed and dated by the analyst to ensure the consistency and accuracy of the data produced. Refer to Figure 1.0 for the QCAR and specific control points covered.

11.8.2 Specific quality control check points have been established for the preparation of data deliverables which are monitored through a Quality Control Approval Report (QCAR). The specific check points must be initialed and dated by the analyst to ensure the consistency and accuracy of the data produced. Refer to Figure 1.1 for the QCAR and specific control points,covered.

11.9 Analytical Documentation Procedures

11.9.1 Instrument batches

An instrument batch is created for each analytical sequence to organize all the associated data. Batch designations are of the format:

Xnnnn

where

X = instrument identifier

nnnn = number of batch

(i.e. A0012)

Instrument batches are number sequentially so each analytical sequence is identified by a unique batch identifier. The batch consists of a file folder wit all the associated QC information for the analytical sequence. The raw data is then bound together with the file folder to complete the batch.

11.9.2 Filing system

All active batches are filed chronologically according to instrument. The batches are transferred to file boxes for long term storage once all the associated data within a batch has been completed.

11.9.3 Data archiving

All data files, including the BFB analysis data file, are archived on a real time basis using the ChemStation back-up system. Care shall be exercised when purging data off the hard drives to ensure that all data being purged has been archived. This is checked by producing an archive directory on an analytical batch basis, verify that the data associated with that batch has been properly archived. If files are not found on the archive listing, then those files must be manually archived and a new listing produced. The daily archive directories are checked and filed in each associated batch.

If files have been queued for archiving and it is becomes necessary to delete them from the archive list, then the files deleted must be manually archived once the problem has been corrected.

A full archive directory listing is produced when the archive tape being used is full. This directory listing is kept on file in the instrument room.

11.9.4 Instrument run logs

It is STL's policy that all measurement data be recorded in logbooks or on preprinted log sheets in permanent ink. Transcriptions shall be avoided whenever possible. The record shall reflect the measurement performed and all appropriate details for conclusions related to the measurement. The record shall be signed and dated by the individual performing the measurement on the day the measurement is performed. Corrections shall be made by drawing a single line through the error, and initialing and dating the correction. A secondary authorization of the logbook is required and shall be performed by the department's manager or designee.

Each instrument has its own set of bound run logs which are sequentially number and paginated. Run logs are filed in the laboratory once they have been filled, for future reference. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard codes used shall be completed for each sequence. All standards used are recorded in this field for future traceability. The data file, job number, sample number, quantitation factor, dilution factor, analyst's signature, and date are recorded. Refer to Figure 2.0.

11.9.5 Initial data review sheet

The initial data review sheet (IDRS) is a computerized review sheet which is used to check the key quality control criteria for compliance. The IDRS is used to check that all samples

have been analyzed with the required calibration time frame, if the IS's meet RT and EICP area criteria, and if the ID file being utilized was correctly updated. The IDRS is also used as the initial data review tool. Each sample is listed on the sheet and it is either accepted or rejected in the right hand column, by the analyst performing the data review. If reruns are required for dilutions, then the analyst shall indicate the proper dilution required for reanalysis. The data reviewer initial's and dates the IDRS. The batch is then is secondary reviewed by another analyst to ensure that no oversight errors were made during the initial review. The batch is then filed for use during deliverables preparation. Refer to Figure 3.0 for an example of the IDRS.

11.9.6 Corrective action reports

A corrective action report (CAR) is issued when a problem is encountered during analysis, data reduction or deliverables preparation, data validation, or when any deviations from this SOP occur. The CAR is prepared by the analyst first identifying the problem an is then submitted to the department's manager for approval. The manager will give the CAR to the QC officer to be recorded and circulated.

The lower portion of the CAR is for the corrective actions taken, and is completed by the department or project manager when a corrective action decision has been made. The CAR is then redistributed to all the departments and individuals involved. Refer to Figure 4.0.

11.9.7 Chain of custody record

When samples are removed from storage for preparation or analysis they must be singed out utilizing the chain of custody record (COC). The samples shall then be signed back in on the COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation or analysis. Refer to the sample removal SOP, referenced in section 15.0.

12.9.8 Sample tracking record

Samples are tracked on the Volatile Sample Tracking Report (VSTR) upon preliminary notification of their receipt. When samples are analyzed the analyst initial's and dates the VSTR. The VSTR is updated after initial data review, designating that the samples is analyzed and complete with its associated data file, or that the sample requires reanalysis. Samples requiring reanalysis are also documented as to the reanalysis dilution required, if any. Refer to Figure 5.0.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 Instrumental Conditions

12.1.1 Purge & Trap Device

Purge Conditions: (VOACARB)

Purge Gas:	Helium
Purge Time:	11.0 min
Purge Flow:	40 ml/min
Dry Purge:	3.0 min
Purge Temp:	Ambient for LLW and MLS

Desorb Conditions: (VOACARB)

Desorb Temp:	250°C
Desorb Flow:	15 ml/min
Desorb Time:	4.0 min

Trap Reconditioning Conditions: (VOACARB)

Reconditioning Temp:	270°C
Reconditioning Time:	9.0 min

12.1.2 Gas Chromatograph

Carrier Gas:	Helium
Flow Rate:	5 ml/min
Initial Temp.:	30°C
Initial Hold:	4 min
Ramp Rate 1:	5°C/min
Second Temp.:	100°C
Ramp Rate 2:	12°C/min
Final Temp.:	200°C
Final Hold:	1.0 min
Transfer Temp:	185°C

12.1.3 Mass Spectrometer

Electron Energy:	70 eV
Mass Range:	35 - 300 amu

Scan Time: less than 1 sec/scan

The mass spectrometer must be tuned to meet the instrument performance check criteria for 50 ng of BFB listed in Table 3.0.

12.2 Sample Analysis Procedures

12.2.1 Water Samples (using guidance from method 5030B)

Samples are removed from Sample Control storage and are signed out in the chain of custody form. Refer the sample removal from the laboratory SOP, referenced in section 15.0.

All samples are allowed to warm to room temperature.

Make sure all instrumental operating conditions are correctly set and BFB, calibration and blank criteria have been met.

In a gas tight 5 ml syringe, load a 5 ml aliquot of sample (or 25 ml depending on request) by removing the plunger and attaching a closed syringe valve. Open the sample which has been allowed to come to ambient temperature, and carefully pour the sample into the barrel of the syringe until just short of overflowing. Replace the syringe plunger and replace the plunger. Open the syringe valve and vent any residual air, then adjust the volume to 5.0 ml (or 25 ml). Drop a few drops of the sample on narrow range pH paper (0-6pH range) record the pH in the logbook for all aqueous samples. This procedure destroys the integrity of the sample for future analysis, therefore, if there is only one vial, the analyst shall fill a second gas tight syringe in the same manner. This second syringe is maintained only until such time as the analyst has determined that the first sample has been properly analyzed. If an analysis is required from the second syringe, it must be performed within 24 hours. Care must be taken to prevent air from leaking into the syringe during storage.

Next, spike the sample with 5 uL of the IS and SMC 50 ug/ml spiking solution utilizing a 25 uL gas-tight, through the open syringe valve, then load the syringe contents into the sparging vessel. If an autosampler is utilized, set up the autosampler start and stop positions for the sequence being purged.

Inject the sample into the purging chamber and purge at ambient temperature. After purging, the sample is thermally desorbed onto the GC column.

While the trap is being desorbed into the GC, empty the purging chamber either manually or with the autodrain option. Wash the chamber with a minimum of two 5 ml flushes of reagent water to avoid carryover of target compounds.

The trap is then reconditioned while the sample is temperature programmed on the GC to separate the volatile organics.

When a sample has been analyzed that has saturated ions from a compound, this position on the automatic liquid sampler will be capped off and not used until a system blank has been analyzed and deemed clean for that position. If the blank is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank has been analyzed that demonstrates that the system is free of interferences. Once the system is free of interferences, the sample that saturated the detector must be diluted and reanalyzed.

If a sample is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, but not saturated, then the sample must be reanalyzed at an appropriate dilution. The purge and trap system shall be demonstrated to be free from carry-over through the subsequent analyses of blanks and/or samples which do not contain the target compound at a concentration greater than the PQL.

When the system is run unattended, using the autosampler, if a sample is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, then the sample must be reanalyzed at an appropriate dilution. The samples analyzed subsequently shall be carefully evaluated. If any subsequent analyses contains the target compounds which were at concentrations greater than the initial calibration upper limit in the previous sample, at a concentrations greater than the PQL, then those samples must be reanalyzed once the system has been decontaminated and shown to free of interferences.

A water MS/MSD (MSB-NYSDEC) is prepared by spiking the sample aliquot with 5 uL IS and SMC, along with 5 uL of matrix spike solution using a 25 uL gas-tight syringe. The spiked sample is then analyzed as previously described.

A method blank must be analyzed every 12 hours after the calibration criteria has been achieved. The method blank consists of 5 ml reagent water spiked with 5 uL of IS and SMC, and carried through the analytical procedure. Method blank criteria is defined in section 10.3.3.

12.2.2 Low Level Soil Samples (using guidance from method 5035)

The type of sample container that was used by the client must be determined at time of sample receipt. A hermetically sealed voa vial with preservative can be held for 14 days from collection. A sealed soil plug sample such as the EnCore sampler must be either transferred to a voa vial with 5mls of a Sodium Bisulfate solution and a magnetic stir bar, or analyzed within 48 hours from collection.

Samples are removed from Sample Control storage and are signed out on the chain of

custody form. If the soil is received in a sealed plug type sampler such as the EnCore sampler, then the plug is transferred to a pre-weighed voa vial containing 5mls of the Sodium Bisulfate solution and a magnetic stir bar. The vial is sealed and not opened until time of disposal. The vial is then re-weighed and the weight is recorded on the vial along with the subtracted weight that determines the soil weight. The client ID is transferred along with the laboratory ID onto the voa vial. The vials are also numbered for each sample transferred to make each vial unique.(ie vial 1,2 or 3). If QC has been requested on a particular sample the vials are labeled "MS" or "MSD". Once the soil is transferred and the above steps have been performed, the soil is placed in the volatile refrigerator for storage.

All samples are allowed to warm to room temperature.

Make sure all instrumental operating conditions are correctly set and BFB, calibration and blank criteria have been met.

The sample consists of the entire contents of the sample container. The voa vial is not to be opened. This sample is then loaded on the Archon autosampler. The internal standards and surrogates along with 5mls of reagent water will be added automatically by the Archon prior to the sample pre-heat mode. Then the voa vial will be purged onto the Voacarb 3000 trap. Then analyzed by the Mass Spectrometer.

A percent moisture determination is performed by weighing out 5 grams soil from a separate container that has been submitted for percent solid determination. The soil shall dry overnight at 105 degrees Celsius. Allow to the sample to cool before weighing the sample back. The percent moisture is determined according to the following equation:

$$\frac{\text{g of wet sample - g of dry sample}}{\text{g of wet sample}} \times 100$$

The trap is then reconditioned while the sample is temperature programmed on the GC to separate the volatile organics.

If a sample is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, then the high concentration method must be utilized, unless the client sent other sealed voa vials with a lower sample volume that would be within the linear calibration range. Then the sample must be reanalyzed at an appropriate dilution. No less than 1.0 gram of sample may be analyzed by the low level method (i.e. a 5 fold dilution) utilizing the low level soil method. If a larger dilution is required, then the medium level soil method must be employed.

The purge and trap system shall be demonstrated to be free from carryover through the subsequent analyses of blanks and/or samples which do not contain the target compound

at a concentration greater than the PQL.(see section 9.2 for detail)

A low level soil MS/MSD (MSB-NYSDEC) is prepared by spiking a separate sample vial. The voa vial is not to be opened. This sample is then loaded on the Archon autosampler. The internal standards and surrogates along with 5mls of reagent water will be added automatically by the Archon prior to the sample pre-heat mode. Then the voa vial will be purged onto the Voacarb 3000 trap. Then analyzed by the Mass Spectrometer.

A low level soil method blank must be analyzed every 12 hours after the calibration criteria has been achieved. The method blank consists of 5.0 grams of a purified solid matrix added to 5.0 ml of reagent water and a magnetic stir bar. The method blank is then loaded on the Archon and automatically spiked with internal standards , surrogates ,and 5mls of reagent water then carried through the analytical procedure. Method blank criteria is defined in section 10.3.3.

12.2.3 Medium Level Soil Samples

Samples are removed from the Sample Control storage area and signed out on the chain of custody form.

All samples are allowed to warm to room temperature.

Make sure all instrumental operating conditions are correctly set and BFB, calibration and blank criteria have been met.

12.2.3.1 Laboratory Preserved

More than one scenario may take place with high concentration soil samples. Review the client's Quality Assurance Plan prior to preparing samples to ensure there are no other site requirements for preparing the methanol extracts. If the client expected high levels of volatiles in the field, then the samples were collected in the field preserved in methanol. Or the client may use the EnCore sampler or equivalent, therefore the plug of soil from the EnCore sampler is placed into the pre-weighed voa jar containing 10mls of purge and trap grade methanol. Record the weight of the jar, soil and methanol. Record the soil weight on the jar. Shake the jar for 2 minutes, then let the contents settle and transfer 1-2mls of the methanol extract into a extract vial. Store extract until time of analysis. It is also possible that a low level soil is actually a high level soil. Therefore the methanol extraction is performed at the laboratory. If the sample is extracted at the laboratory, then extract as follows:

The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. A medium level soil extract is prepared by weighing out 5.0 grams of sample into a 20 ml extraction vial, record the weight to the nearest 0.1 gram. Determine the percent

moisture as in the low level method. Quickly add 10.0 ml of purge and trap grade methanol to the vial. Cap and shake for 2 minutes.

Using a disposable pipette, transfer about 1 ml of sample extract to a GC vial for storage. The remainder of the extract may be discarded. Transfer about 1 ml of the reagent methanol to a GC vial for use as the method blank. These extracts may be stored in the dark at 4 degrees Celsius (+/- 2 degrees) prior to analysis.

In a gas tight 5 ml syringe, load a 5 ml aliquot of reagent water and spike with 5 uL of IS/Surrogate spiking solution using a 25 uL gas-tight syringe, and 100 ul of sample using a 250 uL gas tight syringe, extract and purge for 11.0 minutes at ambient temperature. If an autosampler is utilized, set up the autosampler start and stop positions for the sequence being purged. After purging, desorb onto the GC column.

The medium level soil extract is analyzed under a water initial and continuing calibration.(using guidance form method 5030B)

The trap is then reconditioned at 220 degrees Celsius for 8 minutes while the sample is temperature programmed on the GC to separate the volatile organics.

If an extract is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, then the extract must be reanalyzed at an appropriate dilution. Volumes of less than 10 ul (i.e. 10 fold MLS dilution) shall be prepared diluting an aliquot of the methanol extract and then taking 100 uL for analysis. Add the volume of methanol extract from the sample and a volume of clean methanol to total 100 ul. The total methanol volume added shall be 100 ul, excluding the methanol in the standards.

The purge and trap system shall be demonstrated to be free from carry-over through the subsequent analyses of blanks and/or samples which do not contain the target compound at a concentration greater than the PQL.

A medium level soil MS/MSD is prepared by spiking a 5.0 gram sample extract with 9.0 ml methanol, along with 1.0 ml of matrix spike solution. The spiked sample extract is then analyzed as previously described.

A method blank must be analyzed every 12 hours after the calibration criteria has been achieved. The method blank consists of 10.0 ml of reagent methanol. A 100 uL aliquot of the method blank extract is then spiked into 5 ml reagent water fortified with 5 uL IS/Surrogate solution. The method blank is then carried through the analytical procedure as described previously. Method blank criteria is defined in section 10.3.3.

12.2.3.2 Field Preserved

If high volatile concentrations are expected in the soil samples a different sampling

method is required. The soil vial will contain methanol, be pre-weighed and labeled, prior to shipping to the client. There will also be an empty soil vial for total solids determination in the laboratory. Five grams of soil will be added to the methanol vial in the field and weights recorded on the chain of custody, vials or other appropriate locations. A permanent mark will be scribed on the vial at the level of the methanol and soil mixture. Oily waste samples will be collected in empty vials (unless known to be soluble in methanol or polyethylene glycol (PEG) * PEG must be requested when placing bottle orders otherwise vials will contain methanol. The laboratory will weigh the voa vial prior to analysis, record the weight in the logbook, check to ensure the methanol and soil mixture is still at the scribe mark. If not a corrective action must be written to notify client of possible re-sampling, or another acceptable solution. The sample is then shaken and allowed to settle. A portion of the diluted sample is then removed from the vial and analyzed by method 5030B.(see section 11.2.1) Total solids, sample weight, and sample volume used, all need to be recorded to determine correct dilution factor for quantitation.

12.3 Qualitative Analysis

12.3.1 Target Compounds

The relative retention time of a target compound must be within +/- 0.06 RRT units of the RRT of the calibration standard for a positive identification. For reference the standard must be analyzed within the same 12 hour time period as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT shall be assigned by using the extracted ion current profiles for ions unique to the component of interest.

In addition, a comparison must be made between the mass spectrum obtained in the sample analysis and the reference mass spectrum for that compound, which was obtained on that specific GC/MS system. The requirements for qualitative verification by comparison of mass spectra are as follows:

All ions present in the reference spectrum at an intensity greater than 10% must be present in the sample spectrum.

The relative intensities of the ions above 10% must agree with 20% between the reference and sample spectra.

Ions greater than 10% in the sample spectrum but not present in the reference spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by the above criteria, but in the technical judgement of the analyst, the identification is correct, then the compound shall be reported.

12.3.2 Tentatively Identified Compounds

If requested a library search shall be performed for non-target compounds in the sample for purposes of tentative identification. For this purpose, the most recent release of the NIST mass spectral library shall be used.

Up to 10 organic compounds of apparent concentration not listed in Table 1.0, shall be tentatively identified via a forward library search. Only compounds with responses greater than 10% of the closest IS exhibiting no interference are to be searched.

The ChemStation software is utilized to perform the automated library search. The program (TICS) is executed with the data file, quantitation file, and number of compounds to be searched, specified for each sample or blank. Prior to running the program, the analyst must delete from the quantitation file, using the QDEL program, the non-TCL compounds which were identified in the quantitation file. This will facilitate their automated search using the program. If the non-TCL positive hits are not removed prior to executing the program, they would be counted as target compounds and not be searched by the program, leading to false negatives.

A tentative identification will be made after a comparison between the mass spectrum obtained in the sample analysis and the library search mass spectra found for that compound. The requirements for tentative verification by comparison of mass spectra are as follows:

Ions present in the reference spectrum at an intensity greater than 10% should be present in the sample spectrum.

The relative intensities of the ions above 10% should agree with 20% between the reference and sample spectra.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or coeluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible background subtraction by the data system.

If in the technical judgement of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound shall be reported as unknown. Additional classification shall be made if possible (i.e. Unknown hydrocarbon).

12.4 Quantitative Analysis

12.4.1 Target Compounds

Target compounds are quantitated by the internal standard technique. The associated internal standard used is listed in Table 6.0. The EICP area of the quantitation ions of

compounds listed in Tables 8.0 and 8.0 are used. The quantitation ion for the SMC compound bromofluorobenzene shall be m/z 174 instead of m/z 95 due the coelution of the target compound 1,1,2,2-tetrachloroethane, which interferes with m/z 95.

The relative response factor (RRF) from the initial calibration standard or the equation for linear regression is used to calculate the concentration in the sample depending on the percent RSD in the calibration curve. When compound concentrations are below the PQL, but the compound meets identification criteria, report the concentration with a "J" qualifier.

Water Samples

Concentration

$$ug/L = \frac{(Ax)(Is)(Df)}{(Ais)(RRF)(Vo)}$$

where,

Ax = area of the compound quantitation ion

Ais = area of IS quantitation ion

Is = IS amount in nanograms

RRF = Average Relative response factor from the ambient temperature purge of the Initial calibration curve (Linear Regression may replace this equation if the mean response for all the compounds in the method is above 15% RSD in initial calibration)

Vo = volume of water purged in ml's

Df = Dilution factor. The dilution factor for analysis of water samples for volatiles by this method is defined as the ration of the number of milliliters (ml) of water purged (i.e. Vo above) to the number of ml of the original water sample used for purging. For example, if 2.5 ml of sample is diluted to 5.0 ml with reagent water and purged, DF=5.0 ml/2.5 ml = 2.0. If no dilution is performed, Df = 1.0.

Low Level Soil Samples

Concentration
(dry weight basis)

$$ug/Kg = \frac{(Ax)(Is)}{}$$

$$(Ais)(RRF)(Ws)(D)$$

where,

Ax, Is, and Ais are as given for water.

$$D = \frac{100 - \%\ moisture}{100}$$

Ws = weight of sample added in grams

RRF = Average Relative response factor from the heated temperature purge of the Initial calibration curve. (Linear Regression may replace this equation if the mean response for all compounds in the method is above 15% RSD in initial calibration)

Medium Level Soil Samples

Concentration
(dry weight basis)

$$ug/Kg = \frac{(Ax)(Is)(Vt)(1000)(Df)}{(Ais)(RRF)(Va)(Ws)(D)}$$

where,

Ax, Is, Ais and RRF are as given for water.

$$D = \frac{100 - \%\ moisture}{100}$$

RRF = Average Relative response factor from the ambient temperature purge of the initial calibration curve. (Linear Regression may replace this equation if the mean response for all compounds in the method is above 15% RSD in initial calibration)

Ws = weight of sample extracted in grams

Vt = total volume of methanol extract in ml

Va = volume of the methanol extract added to the reagent water for purging in ul

Df = Dilution factor. The dilution factor for medium level soils is defined as the ratio of

the number of microliters (uL) of methanol added to the reagent water for purging (i.e. Va above) to the number of uL of the methanol extract of the sample contained in that volume Va. The dilution factor is equal to one in all cases other than those requiring dilution of the methanol extract.

12.4.2 Tentatively Identified Compounds

An estimated concentration for non-target compounds tentatively identified in the sample shall be determined by the internal standard method. For quantitation, the nearest IS free of interferences shall be used.

The equation for calculating concentrations are the same as in 11.4.1. Total area counts from the total ion chromatograms are used for both the IS and compound. A RRF of 1.0 is assumed and the resulting concentration shall be qualified as "J" (estimated), indicating the quantitative and qualitative uncertainties associated with this non-target compound.

12.4.3 The three Xylene isomers are to be reported as Xylenes (total). The meta and para isomers coelute on the capillary column, therefore, special attention must be given to their quantitation. The area from the o-Xylene is then used to quantitate the Xylenes (total) concentration. All three isomers must be present in the initial and continuing calibration standards.

12.4.4 The cis and trans isomers of 1,2-Dichloroethene are to be reported as 1,2-Dichloroethene (total). The two isomers do not coelute on the capillary, therefore, the RRF is determined by summing the two isomer areas and then dividing by the total isomer concentrations. The area from both peaks and this RRF are then used to quantitate the 1,2-Dichloroethene (total) concentration. Both isomers must be present in the initial and continuing calibration standards.

12.4.5 If the on-column concentration of any compound in any sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and purged. Guidance in performing dilutions, and exceptions to this requirement are as follows:

12.4.5.1 Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

12.4.5.2 The dilution factor chosen shall keep the response of the largest analyte peak for a target compound in the upper half of the initial calibration range of the instrument.

12.4.5.3 Data for more than two analyses shall not be submitted.

12.4.5.4 Run associated sample at correct dilution prior to running MS/MSD. It is not nessesary to re-run a MS/MSD if internal standards and or surrogates are out of criteria. Or spike compounds are outside of recovery windows. However; MSB's Must meet Internal

standard and surrogate criteria, and all spike compounds must be within criteria. Note the problem in the SDG narrative.

12.4.5.5 For total Xylenes, where three isomers are quantified as two peaks, the calibration of each peak, should be considered separately, i.e. a diluted analysis is not required for total Xylenes unless the peak representing the single isomer exceeds 200 ug/L or the peak representing the two coleuting isomers exceeds 400 ug/L. Keep in mind that the average RF from o-Xylene must be used to determine if dilution is correct.

12.5 Instrument Maintenance

12.5.1 Preventative maintenance

All HP instrumentation is covered by a service contract with an external instrumentation service vendor, or by STL personnel trained in preventative maintenance. Preventative maintenance is perform at scheduled intervals on all equipment according to the frequency detailed in Appendix A. All instrument preventative maintenance is performed according the manufacturers recommended procedures, by trained personnel. All preventative maintenance shall be thoroughly documented in the maintenance log (see Figure 6.0), as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

12.5.2 Corrective maintenance determinants and procedures

Corrective maintenance is deemed necessary when the analytical system, after reanalysis, can not meet tune, calibration, or other protocol specific QC criteria. Corrective maintenance may include, but is not limited to, decontamination of the system, source cleaning, replacing the electron multiplier, column replacement, jet separator cleaning or replacement, or filament replacement. All corrective maintenance is performed according the manufacturers recommended procedures, by trained personnel. All corrective maintenance shall be thoroughly documented in the maintenance log, as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

12.5.3 Maintenance authorization

All preventative and corrective maintenance is authorized by the department's manager, or designee. When maintenance is deemed necessary, a service call is placed for all equipment covered under a service contract, by the department's manager, or designee.

12.6 Data System

12.6.1 Data Acquisition and System Operation

Data is acquired from sample analyses using the ChemStation software. Analytical batches are set up with all the associated sample ID, dilution, and data file information. Automated post-acquisition quantitation is qued with the appropriate method, as well as, post-acquisition archiving of the data file. The sequence is assigned and started using the ChemStation software.

12.6.2 Instrument errors

System errors are logged to the ChemStation error logs. The system manager shall be responsible for checking and providing corrective actions for all major system errors. Minor system errors, such as insufficient disk space, are handled by trained analysts, as necessary.

12.6.3 Manual Integrations and Editing Flags

Manual integrations are required when the automated software doesn't correctly integrate extracted ion current profiles (EICP). Manual Integrations are performed. The target compound number of interest is selected and the EICPs are graphically presented. The peak can then be correctly integrated. A new quantitation report is produced. The manually integrated data file is the saved by exiting and saving from file edit. Manual integrations are flagged by the data system with the "M" qualifier beside any manually integrated area on the hardcopy quant report. The analyst shall initial and date the hardcopy report for the manual integrations performed.

13.0 CALCULATIONS

13.1 Relative Response Factor (RRF)

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

where,

Ax = area of the compound quantitation ion

Ais = area of IS quantitation ion

Cis = IS concentration

Cx = compound concentration

An average RRF is calculated for each compound and SMC from the initial calibration.

The RRF used for quantitation is based upon the ortho-Xylene isomer peak. The area from both peaks and this RRF are then used to quantitate the Xylenes (total) concentration.

All three isomers must be present in the initial and continuing calibration standards.

The RRF is used for 1,2-Dichloroethene is calculated by summing the two isomer areas and then dividing by the total isomer concentrations. The area from both peaks and this RRF are then used to quantitate the 1,2-Dichloroethene (total) concentration in a sample. Both isomers must be present in the initial and continuing calibration standards.

13.2 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{\text{standard deviation}}{\text{mean}} \times 100$$

13.3 Percent Difference (%D)

$$\%D = \frac{(\text{average RRFi}) - (\text{RRFc})}{(\text{average RRFi})} \times 100$$

where,

average RRFi = average RRF from the initial calibration

RRFc = RRF from the continuing calibration standard

13.4 Percent Moisture

$$\%\ \text{moisture} = \frac{\text{g of wet sample - g of dry sample}}{\text{g of wet sample}} \times 100$$

13.5 Target Compound Concentrations

The calculations used to determine the target compound concentrations are described in section 11.4.

13.6 SMC Percent Recovery

$$\%\ \text{Recovery} = \frac{\text{concentration found}}{\text{concentration spiked}} \times 100$$

13.7 Matrix Spike Recovery

$$\%\ \text{Recovery} = \frac{SSR - SR}{SA} \times 100$$

where,

SSR = spiked sample result

SR = sample result

SA = spike added

13.8 Relative Percent Difference

$$RPD = \frac{\text{absolute (MSR - MSDR)}}{(½)(MSR+MSDR)} \times 100$$

where,
MSR = matrix spike recovery

MSDR = matrix spike duplicate recovery

The absolute value of the recovery difference is used in the above equation.

13.9 Adjusted Contract Required Quantitation Limit for Samples

$$\text{Adjusted PQL} = \frac{(PQL) \times Df}{D}$$

where,

$$D = \frac{100 - \%\ \text{moisture}}{100}$$

Df = the dilution factor

14.0 ACCEPTANCE OF DATA

14.1 Method Blank

The method blank must contain one half, or less of the PQL of the target compounds, except methylene chloride and acetone which must be less than or equal to three times the PQL. Special projects may require lower detection limits than the PQL. In these cases the method blank must not contain compounds over the client requested detection limit.

If a method blank exceeds the limits for contamination above, the laboratory shall consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds.

14.2 System Monitoring Compounds (SMC)

All SMC's must be within the recovery criteria listed in Table 5.0. Method blanks and samples with recoveries outside the required windows must be reanalyzed. Refer to section 10.6 for SMC information.

14.3 Instrument Performance Check

The criteria for bromofluorobenzene is listed in Table 3.0 and in section 10.3.1.

14.4 Internal Standards
The IS criteria is described in section 10.7.

14.5 Matrix Spike/Matrix Spike Duplicate

The MS/MSD criteria is described in section 10.4.

15.0 REPORTING OF RESULTS

Refer to documentation policy/procedures organics SOP and the data reduction, management, and handling procedures SOP, referenced in section 15.0.

16.0 POLLUTION PREVENTION

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 WASTE MANAGEMENT

17.1 All waste shall be managed in accordance with all state and federal requirements. See the STL-CT Hazardous waste management plan.

17.2 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 Standard Operating Procedure Documentation Policy/Procedures Organics, 15.2SOP for Volatile Standards Preparation

18.3 SOP for Samples Processing Methods Performed at Sample Arrival

18.4 SOP for Log-In Methods for CLP Samples

18.5 SOP for Storing Water and Soil Samples for Organic and Inorganic Sample Analysis

18.6 SOP for Documenting Sample Removal from the Laboratory

18.7 Standard Operating Procedure for Sample Tracking

19.0 REFERENCES

19.1 Purge & Trap Method EPA SW846 3rd Edition, Methods 5030B/5035.

19.2 Volatile organics by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 3rd Edition, Method 8260B.

19.3 EnChem, Inc 1795 Industrial Drive, Green Bay WI 54302 (920) 469-2436
EnChem produces the EnCore sampler a tool that one manually inserts into the soil to take a plug of soil sample. The sample is then capped and shipped to the laboratory. The laboratory then must remove the soil and analyze by either the 5035 soil method or a methanol extraction method (5030B) * soil samples must be analyzed within 48 hours after collection, or transferred to vials. (section 6.2.1.8 5035-10)

19.4 Associated Design and Manufacturing Company, 814 North Henry Street, Alexandria, VA 22314 (703) 549-5999

Purge and Trap Soil Sampler - (model 3780PT) this tool allows a sampler to take a soil sample plug and through the use of various adapters for the voa vial, put the plug of soil into a standard 40ml voa vial, then ship the voa vial and voa vial adapter to the laboratory for analysis by method 5035.

19.5 Becton Dickinson & Co Franklin Lakes NJ 07417-1884 (888) 237-2762 or VWR, 800) 932-5000 Sterile syringe (not cut). The end of the plastic syringe barrel is cut off, either in the field, or by the laboratory prior to shipping. The syringe is tarred out in the field on a portable balance prior to scooping up a sample. Five grams of soil sample is then collected in the syringe and put into a voa vial that has been pre-weighed, preserved with sodium bisulfate and contains a Teflon coated stir bar(all supplied by laboratory). Vials are sealed and samples are directly loaded onto an autosampler which pierces the septa of the voa vial therefore keeping the sample sealed and not opened by the laboratory. Four other vial should be collected in the same manner to ensure enough sample volume for repeated analyses at the laboratory. One empty voa vial should also be filled with soil for total solids determination, and to be used if the soil collected must be run as a medium level soil.

20.0 SUBSTANTIVE REVISIONS

20.1 Original issue; lab update to method 8260B - January 27, 1998.

20.2 Revision on: November 17, 1998. Changed Laboratory name in entire document from AEN to STL. Section 7.0; updated instrumentation and computer systems. Section 11.0; added method 5035 low level soil procedure.

20.3 Corrected section 9.4.1 item number 2 to state "below 15%". Corrected header problem. - January 27, 1999.

20.4 Updated to reflect A.C.O.E. modifications March 19,1999
20.5 Updated to reflect NELAP added sections 3,16,17 renumbered SOP October 6, 1999
20.6 Updated after Laboratory Manager's review 11/05/1999

TABLES

TABLE 1.0
TARGET COMPOUND LIST (TCL) AND ESTIMATED QUANTITATION LIMITS (EQL)

Volatile Organics	Quantitation Limits*			
	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column ng
Chloromethane	10	10	1,000	(50)
Bromoethane	10	10	1,000	(50)
Vinyl Chloride	10	10	1,000	(50)
Chloroethane	10	10	1,000	(50)
Methylene Chloride	10	10	1,000	(50)
Acetone	10	10	1,000	(50)
Carbon Disulfide	5	5	500	(50)
1,1-Dichloroethene	5	5	500	(50)
1,1-Dichloroethane	5	5	500	(50)
1,2-Dichloroethene (total)	5	5	500	(50)
Chloroform	5	5	500	(50)
1,2-Dichloroethane	5	5	500	(50)
2-Butanone	10	10	1,000	(50)
1,1,1-Trichloroethane	5	5	500	(50)
Carbon Tetrachloride	5	5	500	(50)
Bromodichloromethane	5	5	500	(50)
1,2-Dichloropropene	5	5	500	(50)
cis-1,3-Dichloropropene	5	5	500	(50)
Trichloroethene	5	5	500	(50)
Dibromochloromethane	5	5	500	(50)
1,1,2-Trichloroethane	5	5	500	(50)
Benzene	5	5	500	(50)
trans-1,3-Dichloropropene	5	5	500	(50)
Bromoform	5	5	500	(50)
4-Methyl-2-pentanone	10	10	1,000	(50)
2-Hexanone	10	10	1,000	(50)
Tetrachloroethene	5	5	500	(50)
Toluene	5	5	500	(50)
1,1,2,2-Tetrachloroethane	5	5	500	(50)
Chlorobenzene	5	5	500	(50)
Ethylbenzene	5	5	500	(50)
Styrene	5	5	500	(50
Xylene (total)	5	5	500	(50)

*Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

TABLE 2.0
APPENDIX IX COMPOUND LIST AND ESTIMATED QUANTITATION LIMITS (EQL)

Volatile Organics	Quantitation Limits*		
	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg
Chloromethane	10	10	1,000
Bromomethane	10	10	1,000
Vinyl Chloride	10	10	1,000
Chloroethane	10	10	1,000
Methylene Chloride	5	5	500
Acetone	10	10	1,000
Carbon Disulfide	5	5	500
1,1-Dichloroethene	5	5	500
1,1-Dichloroethane	5	5	500
1,2-Dichloroethene (total)	5	5	500
Chloroform	5	5	500
1,2-Dichloroethane	5	5	500
2-Butanone	10	10	1,000
1,1,1-Trichloroethane	5	5	500
Carbon Tetrachloride	5	5	500
Vinyl Acetate	10	10	1,000
Bromodichloromethane	5	5	500
1,2-Dichloropropane	5	5	500
cis-1,3-Dichloropropene	5	5	500
Trichloroethene	5	5	500
Dibromochloromethane	5	5	500
1,1,2-Trichloroethane	5	5	500
Benzene	5	5	500
trans-1,3-Dichloropropene	5	5	500
Bromoform	5	5	500
4-Methyl-2-Pentanone	10	10	1,000
2-Hexanone	10	10	1,000
Tetrachloroethene	5	5	500
1,1,2,2-Tetrachloroethane	5	5	500
Toluene	5	5	500
Chlorobenzene	5	5	500
Ethylbenzene	5	5	500
Styrene	5	5	500

*Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

TABLE 2.0 (continued)
APPENDIX IX COMPOUND LIST AND ESTIMATED QUANTITATION LIMITS (EQL)

Volatile Organics	Quantitation Limits*		
	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg
Xylene (total)	5	5	500
Dibromomethane	10	10	1,000
1,2-Dibromoethane (EDB)	10	10	1,000
1,1,1,2-Tetrachloroethane	10	10	1,000
1,2,3-Trichloropropane	10	10	1,000
Dichlorodifluoromethane	10	10	1,000
Iodomethane	10	10	1,000
3-Chloro-1-Propene	10	10	1,000
2-Methyl-2-Propenenitrile	10	10	1,000
2-Chloro-1,3-Butadiene	10	10	1,000
Methyl Methacrylate	10	10	1,000
Ethyl Methacrylate	10	10	1,000
1,4-Dichloro-2-Butene	10	10	1,000
1,2-Dibromo-3-Chloropropane	10	10	1,000
Acrolein	100	100	10,000
Acrylonitrile	35	35	3,500
Trichlorofluoromethane	10	10	1,000
Pentachloroethane	5	5	500

***Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.**

TABLE 3.0
GC/MS PERFORMANCE STANDARD
BROMOFLUOROBENZENE (BFB)

m/z	Ion Abundance Criteria	% Relative Base Peak	Abundance Appropriate Peak
50	15-40% of mass 95	25.60	25.60
75	30-60% of mass 95	54.84	54.84
95	Base peak, 100% relative abundance	100.00	100.00
96	5-9% of mass 95	7.58	7.58
173	Less than 2 percent of mass 174	0.00	0.00
174	Greater than 50% of mass 95	90.01	90.01
175	5-9% of mass 174	6.66	7.40
176	95-101% of mass 1 74	88.81	98.66
177	5-9% of mass 175	6.52	7.32

TABLE 4.0
MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Compound	% Recovery Water	RPD Water	% Recovery Soil	RPD Soil
1,1-Dichloroethane	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

TABLE 5.0
SYSTEM MONITORING COMPOUND RECOVERY LIMITS

Compound	% Recovery Water	% Recovery Soil
Toluene-d_8	88-110	81-117
Bromofluorobenzene	86-115	74-121
1,2-Dichloroethane-d_4	76-114	70-121

TABLE 6.0
VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SYSTEM MONITORING COMPOUNDS ASSIGNED FOR QUANTITATION

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d_5
Chloromethane	1,1,1-Trichloroethane	2-Hexanone
Bromomethane	Carbon Tetrachloride	4-Methyl-2-Pentanone
Vinyl Chloride	Bromodichloromethane	Tetrachloroethene
Chloroethane	1,2-Dichloropropane	1,1,2,2-Tetrachloroethane
Methylene Chloride	trans-1,3-Dichloropropene	Toluene
Acetone	Trichloroethene	Chlorobenzene
Carbon Disulfide	Dibromochloromethane	Ethylbenzene
1,1-Dichloroethene	1,1,2-Trichloroethane	Styrene
1,1-Dichloroethane	Benzene	Xylene (total)
1,2-Dichloroethene (total)	cis-1,3-Dichloropropene	Bromofluorobenzene (smc)
Chloroform	Bromoform	Toluene-d_8 (smc)
1,2-Dichloroethane		
2-Butanone		
1,2-Dichloroethane-d_4 (smc)		

(smc) - system monitoring compound

TABLE 7.0
CHARACTERISTIC IONS FOR SYSTEM MONITORING COMPOUNDS AND INTERNAL STANDARDS FOR VOLATILE ORGANIC COMPOUNDS

System Monitoring Compounds	Primary Ion	Secondary Ion(s)
4-Bromofluorobenzene	95	174, 176
1,2-Dichloroethane-d_4	65	102
Toluene-d_8	98	70, 100

Internal Standards	Primary Ion	Secondary Ion(s)
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene-d_5	117	82, 119

TABLE 8.0
CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS

Analyte	Primary Ion*	Secondary Ion(s)
Chloromethane	50	52
Bromomethane	94	96
Vinyl Chloride	62	64
Chloroethane	64	66
Methylene Chloride	84	49, 51, 86
Acetone	43	58
Carbon Disulfide	76	78
1,1-DIchloroethene	96	61, 98
1,1-Dichloroethane	63	65, 83, 85, 98, 100
1,2-Dichloroethene	96	61, 98
Chloroform	83	85
1,2-Dichloroethane	62	64, 100, 98
2-Butanone	43**	57
1,1,1-Trichloroethane	97	99, 117, 119
Carbon Tetrachloride	117	119, 121
Bromodichloromethane	83	85
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166
1,2-Dichloropropene	75	77
Trichloroethene	130	95, 97, 132
Dibromochloromethane	129	208, 206
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Benzene	78	---
cis-1,3-Dichloropropene	75	77
Bromoform	173	171, 175, 250, 252, 254, 256
2-Hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58, 100
Tetrachloroethene	164	129, 131, 166
Toluene	91	92
Chlorobenzene	112	114
Ethylbenzene	106	91
Styrene	104	78, 103
Total Xylenes	106	91

* The primary ion should be used unless interferences are present, in which case, a secondary ion may be used.
** m/z 43 is used for quantitation of 2-butanone, but m/z 72 must be present for positive identification.

FIGURES

FIGURE 1.D

Severn Trent Laboratories

200 Monroe Turnpike
Monroe, Connecticut, 06468

QUALITY CONTROL APPROVAL REPORT

GC/MS Analysis Checklist

QC Batch_____

Chain of Custody forms have been completed.		
Initial Calibration meets the following criteria: ( )CLP3.1 ( )CLP4.1 ( )NYS95 ( )SW846 ( )Other		
Continuing calibration meets the following criteria: ( )CLP3.1 ( )CLP4.1 ( )NYS95 ( )SW846 ( )Other		
Retention times and areas have been verified in the standards.		
RR files generated for all Standards		
VSUM criteria met for all accepted analysis: Internal Standards Surrogates - All accepted samples in clock		
Form III generated (if required), reviewed for outliers		
QCS recovery results reviewed , copied and filed.		
All raw data is present with correct header information and quantitation factors. Areas have been verified.		
Required TIC's have been queued for generation.		
RR files generated for all Samples and reviewed on SeedPak.		
Sample tracking/breakdown sheets have been entirely updated for all batch results.		
All required TIC's have been generated. TICs have been checked for graphics and completion.		
Curve updated on SeedPak.		
QC batch folder is complete per SOP.		
Production log has been updated.		
All samples qedited and chromatograms signed		
Manual integrations double signed and EICP printed.		
Corrective actions submitted (as needed).		

This data meets the requirements of the GC/MS SOP's, unless otherwise documented in a Corrective Action Report.

_____Date_____

Authorizing Signature

DOC.# MSF00204.CT

FIGURE 1.1-8

LMD 03/18/99

STL-CT

GC/MS Quality Control Approval Report

Deliverables: GC/MS Volatiles:_____ Semivolatiles:_____

Project No.:_____ Client:_____

Case:_____ SDG:_____ Deliverables Requested:_____

	Initial Approval Initials/Date	Final Approval Initials/Date
1. The analysis QCAR's are complete		
2. Client & Lab ID's Match		
3. Raw Data has been sorted		
4. All samples & QC present		
5. All raw data present and legible		
6. All required forms completed & Qualifiers verified		
7. Injection Log/Summary copies present		
8. IDL's are present		
9. Narrative complete		
10. Chronicale/NYSDEC forms present		
11. Diskette generated & present		
12. Results transcriptions checked		
13. Holding times meet criteria		
14. Deliverables in correct order		
15. Extraction log/COC present (BNA)		
16. Tabulars generated		
17. QC Outliers noted in narrative		
18. Corrective Action Reports copied and reported As needed		

Deliverables Feedback:

This data is complete as per the requirements of the GC/MS SOP's unless otherwise documented in the narrative.

Final Approval:_____Date:_____

FIGURE 2.0

GC/MS VOLATILES INJECTION LOG Instr. HP5995G IEA/CT

Standards Codes:	Routine Maintenance Performed:	Date: 12/30/93
P:122993:48C:MS2	P:120193:46C:MS2	QC Batch: G2275
P:121493:44C:MS2		IDfile: I-GW Qcaled@ 11:10
P:122193:50A:MS2	P:120193:46B:MS2	Calib file: C-GW
P:120193:45B:MS2		Method file:

Calibrated for the following (please check):
Protocol= SW846( ) CLP 1/91( ) NYS91( ) 524.2 ( ) Other( )
List= Tal( ) PP( ) 524.2 ( ) Other( )

Data file	Client/ Job#	Sample ID	Inj. Time	ALS #	DF	QF	Analyst	Comments
>GB985		50ng 4BFB	09:11	2	1	1		LHP 12 ks/ks
>G9652		VSTD050		2	1	1		
>G9653		Butadiene STD		2	1	1		
>G9654		VBLKG3		2	1	1		
>G9655		VBLKG3		2	1	1		
>G9656	Chem-H 3093-1340	1340095		2	1:250	250		
>G9657		1340094		2	1:100	1:100		
>G9658		1340093		2	1:2500	1:2500		
>G9659		1340092		2	1:5000	1:5000		
>G9660	G+M AUXNP 3093-1420	1420006MS		1	STR	1		
>G9661		1420004MSD		2	STR	1		
>G9662		1420005		5	1:2	2		
>G9663		1420006		6	1:10	5		LHP 12/30/93
>G9664		1420007		12	1:250	250		
>G9665		1420008		13	STR	1		
>G9666		1420009		14	1:1000	1000		
>G9667		50ppb						
>								
>								
>								

Witnessed by: Bobby Date: 01/07/94

Tune File: C:\HPCHEM\MSL\LB326.d
Tune Time: 4 Jan 99 4:07 pm

Reviewed By: LMC Date: 1/5/99
Checked By: _____ Date: _____

Cal File : C:\HPCHEM\MSL\L2448.D

(BCM)	(DFB)	(CBZ)	(DCE)	(TOL)	(BFB)
281450	1.64853e+01	2.22863e+0			

File	Sample	IS Information			Surr Recoveries			
L2449.D	VBLKL6	287350	1660099	1277129	96	105	106	Y
L2450.D	982857A-07	287808	1699319	1255658	98	109	106	Y
L2451.D	982857A-08	291484	1716885	1323323	98	103	104	Y
L2452.D	982825A-06	288661	1701279	1314895	102	105	102	Y
L2453.D	982825A-07	292317	1726129	1330548	104	102	104	Y
L2454.D	982836A-03	283366	1665787	1274823	102	105	105	Y
L2455.D	982836A-05	286002	1670292	1302693	100	105	100	Y
L2456.D	982836A-06	285437	1675269	1300895	102	102	103	Y
L2457.D	982836A-12	282280	1656813	1293124	102	104	98	Y
L2458.D	982836A-15	276111	1621629	1269805	100	103	99	Y
L2459.D	982825A-01	276072	1647500	1266814	103	103	103	Y
L2460.D	982825A-02	291892	1596332	1223267	93	105	100	RR 1:20
L2461.D	982825A-03	290552	1574482	1183237	94	104	102	RR 1:50
L2462.D	982825A-04	267563	1604754	1227152	104	104	103	Y
L2463.D	982825A-05	257105	1505447	1201296	95	104	99	Y

* - fails criteria t - fails 12hr time check

STL

CORRECTIVE ACTION FORM

A. Originator Information

Client Inquiry____

Client:_____ Job/Case:_____

Date/time:_____ Sample Number(s):_____

Client/Lab Contact: _____ Date/Time Response Due:_____

Detailed Description of Potential Problem:_____

_____

_____

_____

_____

_____

_____

_____

_____

B. Quality Assurance Information

Corrective Action ID# _____

Recommended Corrective Action:_____

_____

_____

_____

_____

Groups Involved:

__Sample Control __ Wet Chemistry __ Metals

__Gas Chromatography __ Mass Spectrometry __ Report Generation

__Client Service __ Sample Preparation __Systems ___ Subcontractor

C. Final Resolution

Describe What Happened and Corrective Action Taken:_____

_____

_____

_____

_____

Supervisor Signature: _____ Date_____ Date/Time Client Notified:_____

D. Quality Assurance Final Approval (QA Manager use only)

Corrective Action Approved: _____

Date Finalized: _____

IEA-CT

Figure 5.0

Holding Time Uo	CLIENT	Job Number	Sample Number	Matrix	Date Recv..	Date Collect.	Date Extr.	Date Due	RUSH Y/N	Protocol	Deliv.	Target List	TICS Y/N	MS Batch	1st Run	Rerun Y/N	2nd Run	Rerun Y/N	DATA FILE to Report	DATA FILE to Report RE	CONC
00/00/00	EPA	0001	001	WATER	00/00/00	00/00/00	N/A	00/00/00	Y	CLP3/90	CLP 3/90	TCL	Y						>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	
																			>	>	

FIGURE 6.0

TITLE GC/MS Maintenance Log HP5970H

Project No.

Book No. 1

COP

From Page No.

9/09 Installed new RTX-5 for conditioning @ 20.00 → D Mann

9/23 Installed new RTX-5 for conditioning. Cleaned source (including lens stack). Cleaned ceramics. Changed both filaments → D Mann

9-25 Poor sensitivity in MT.
Found poor vacuum in MSD
Replaced pump oil in RP.
Fys checked OK.

9/28 Replaced filaments, cleaned source (11) Put in new RTX-5 (pre-conditioned 15) → D Mann

10/22 Cleaned source, Cut detector end of column → D Mann

11/09 Removed column, installed new J&W DB-5 column for conditioning. Cleaned source → D Mann

11/19 Cleaned Source - Dave - Conditioned + replaced column on 11/ (PTG-5) - New filaments

11/27 Installed new Restek RTX-5 for conditioning, left overnight @ 300°C

To Page N

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	

APPENDIX A

1.0 Hewlett Packard 5995 GC/MS

Preventative Maintenance	Frequency
*Check oil level in mechanical pumps	Weekly
*Check water level and operating condition in the Neslab cooling units	Weekly
*Check Compressed Air gas supply	Daily
*Check Helium gas supply	Daily
*Check Carbon Dioxide gas supply	Daily
*Change the oil in the mechanical pumps	Every 6 months
*Inspect the pump hoses and replace if required	Every 6 months
*Change oil in the diffusion pump	Every 6 months
*Change foreline and exhaust trap absorbent	Every 6 months
*Inspect and refill the calibration sample vial with PFTBA	Every 6 months
*Vacuum fan grills and filters	Every 6 months
*Check fore and separator pump pressures	Weekly

1.0 Hewlett Packard 5995 GC/MS (continued)

Routine Maintenance	Frequency
*Ion source cleaning and filament replacement	As needed
*Column replacement and conditioning	As needed
*Column cutting and reinstallation	As needed
*Manual tuning	As needed
*Change Compressed Air gas supply	As needed
*Change Helium gas supply	As needed
*Change Carbon Dioxide gas supply	As needed
*Recharge Neslab cooling units	As needed
*Replace electron multiplier	As needed
*Remove and clean or replace jet separator	As needed

2.0 Hewlett Packard 5970 MSD

Preventative Maintenance	Frequency
*Check oil level in mechanical pumps	Weekly
*Change the oil in the mechanical pumps	Every 6 months
*Inspect the pump hoses and replace if required	Every 6 month
*Change oil in the turbo pump	Every 6 months
*Change exhaust trap absorbent	Every 6 months
*Inspect and refill the calibration sample vial with PFTBA	Every 6 months
*Vacuum fan grills and filters	Every 6 months

Routine Maintenance	Frequency
*Ion source cleaning and filament replacement	As needed
*Manual tuning	As needed
*Replace electron multiplier	As needed
*Clean out transfer line to GC	After every column removal

3.0 Hewlett Packard 5890 GC

Preventative Maintenance	Frequency
*Check Helium gas supply	Daily
*Change split vent trap	Every 3 months

Routine Maintenance	Frequency
*Column replacement and conditioning	As needed
*Column cutting and reinstallation	Daily or as needed
*Change Helium gas cylinder	As needed
*Change liner and septum	Daily or as needed
*Clean injection port	As needed

4.0 Hewlett Packard 7672A Autosampler

Preventative Maintenance	Frequency
*Inspect and correct injector alignment	After reseating
*Inspect syringe	Daily
*Check Compressed Air gas supply	Daily
*Inspect and adjust tension on sample tray	Daily
*Change rinse vials	Daily
*Change waste vials	Weekly

Routine Maintenance	Frequency
*Replace syringe	As needed
*Sand injector post	As needed
*Realign autosampler on brackets	As needed
*Change Compressed Air cylinder	As needed

5.0 Hewlett Packard 7673A Autosampler

Preventative Maintenance	Frequency
*Inspect syringe	Daily
*Inspect seating of injector	Daily
*Change rinse vials	Daily
*Change waste vials	Weekly

Routine Maintenance	Frequency
*Replace syringe	As needed
*Reset control box	As needed

6.0 Tekmar Purge and Trap Sample Concentrators and Autosamplers

Preventative Maintenance	Frequency
*Inspect spargers and fittings	Daily
*Check purge flow	Daily
*Inspect line and valve temperatures	Daily

Routine Maintenance	Frequency
*Change and condition trap	As needed
*Adjust purge flow	As needed
*Rinse or clean sparging vessels	As needed
*Rinse sample lines	As needed
*Bake out trap	After each analysis extend as needed
*Replace lines and fittings	As needed
*Adjust line and valve temperatures	As needed

CONTROLLED DOCUMENT REGISTER

DOCUMENT NAME: SOP for GC/MS Volatiles 8260B

ORIGINAL DOCUMENT NUMBER: MS502800.CT

ISSUE #	ISSUED DOC#	RECIPIENT NAME	ISSUED BY	DATE ISSUED
1	MS502800.CT	Marie Vida	M. Cuhl	3/4/98
2	↓	Larry Decker	↓	↓
1	MS502801.CT	Marie Vida	M. Cuhl	11/18/98
2	↓	Larry Decker	M. Cuhl	11/18/98
1	MS502802.CT	Marie Vida	M. Cuhl	1/27/98
2		Larry Decker	↓	↓

8/26/

AEN Form# QAF01000.CT

Severn Trent Laboratories - Connecticut

SOP for the Determination of Trace Metals by ICP-AES
MES02003.CT
Revision 1.0
Date Effective: 01/20/2003

This documentation has been prepared by Severn Trent Laboratories (STL) solely for STL's own use and the use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to Severn Trent Laboratories upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENTCONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF SEVERN TRENT LABORATORIES IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY SEVERN TRENT LABORATORIES IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2003 SEVERN TRENT LABORATORIES, INC. ALL RIGHTS RESERVED.

This is a controlled Document if Stamped in Red:

CONTROLLED COPY # 1

Issued to: Marie Vida

1.0 APPROVALS

The signature of the following individuals indicates that this Standard Operating Procedure (SOP) is complete and meets the requirements specified in the SOP for SOPs.

Laboratory Director

Quality Assurance Manager

Group Leader

2.0 SCOPE AND APPLICATION

2.1 This SOP defines the analysis of samples by inductively coupled argon plasma for samples of both solid and aqueous matrix. Sample is introduced into a plasma where it emits light, which is measured, and the concentrations of analytes is determined.

2.2 The element determined is defined as "Total Metals" for a digested sample. "Dissolved metals" are filtered samples and can be analyzed without digestion.

2.3 The analytes determined using this procedure are:

Aluminum	Cobalt	Potassium	Zinc
Antimony	Copper	Selenium	Boron
Arsenic	Iron	Silver	Silicon
Barium	Lead	Sodium	Strontium
Beryllium	Magnesium	Tin	
Cadmium	Manganese	Titanium	
Calcium	Molybdenum	Thallium	
Chromium	Nickel	Vanadium	

2.4 The document control number for this SOP is MES02003.CT.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used within the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used within the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 **This method is used to analyze samples that have been previously digested for ICP**

analysis. Samples are introduced into a cross flow nebulizer and spray chamber using a peristaltic pump. As the sample enters the plasma it is desolvated, ionized and emits light at characteristic wavelengths for each element. These wavelengths are dispersed by a polychrometer and the intensity measured by a photomultiplier tube. As these intensities are proportional to the concentration these values are compared to a standard curve and the concentration of each element determined.

4.2 This method is based on SW846 method 6010B.

4.3 This method does not deviate from the SW846 method 6010B.

5.0 INTERFERENCES

Spectral interferences include overlaps from other elements, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena and background from stray light or high concentration elements. The first is resolved by IEC's and the last two by background correction. Physical interferences are associated with sample viscosity or high dissolved solids. These are minimized by using a peristaltic pump and/or dilution. Deposits on the torch tip from high solids can be minimized by using a mass flow controller. Chemical interferences are not normally pronounced in ICP and can be minimized by selection of proper operating conditions.

Backround emission and stray light can be compensated for by subtracting the backround emission determined by measurements adjacent to the analyte wavelength peak. To determine the appropriate location for off-line backround correction scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. The interference is expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100-1000 mg/L of the interference element. Spectral interferences are determined by using analyte concentrations that will adequately describe the interference. Interelement correction factors are verified every six months for all analytes. A computer routine is utilized for automatic correction. If the correction routine is operating properly, the determined analyte concentrations from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by ten. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than ten percent should be suspected. The cause of the change is determined and corrected and the correction factor updated.

When interference corrections are applied, their accuracy is verified daily by analyzing spectral interference check solutions.

6.0 SAFETY

Care should be used when working with concentrated acids and all protective gear worn. Safety glasses, lab coat and latex gloves are recommended to protect against glass breakage and sample spillage.

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 SAMPLE CONTAINERS: Plastic, 500 mL capacity or more. Sample bottles are not to be reused.

7.2 SAMPLE COLLECTION: Samples are collected in plastic bottles with nitric acid as a preservative to a pH of less than two.

7.3 SAMPLE PRESERVATION: Sample preservation is with nitric acid as noted above for this method.

7.4 HOLDING TIMES: Samples are analyzed within 180 days of collection.

8.0 APPARATUS AND MATERIALS

8.1 Thermo Jarrell Ash (TJA) 61E Trace ICAP

8.2 Mass Flow Controller

8.3 Peristaltic Pump

8.4 High purity argon, welders grade or better

8.5 Thermo Jarrell Ash Autosampler Model AS 300

8.6 IBM AT Personal Computer and 5.25 floppy diskettes

8.7 1,000 uL variable Eppendorf pipette

8.8 Serological pipettes

8.9 10 mL capacity polypropylene test tubes (Borosilicate glass must not be used for the analysis of Boron or Silicon in order to avoid sample contamination.

8.10 Thermo Jarrel Ash Operator's Manual - February 1991 Part #128832.01

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Reagent water - ASTM Type II, 18 megaohm, deionized

9.2 Trace grade hydrochloric acid and nitric acid.

9.3 ICAP working standards as described in the ICAP standards logbook.

9.4 EPA QC standard solutions or equivalent.

10.0 CALIBRATION

10.1 Calibration standards are prepared from stock solutions and described in labnet data system.

10.2 The calibration is performed in the following manner:

A calibration blank, nano water with 5% nitric acid, is run with the standards prepared to establish a curve which is stored in the computer. A multilevel curve is run along with an ICV (Initial Calibration Verification). Analysis of the ICV must verify that the instrument is within 10% of calibration with a relative standard deviation <5% from three replicate burns(minimum of two) integrations. If the calibration cannot be verified within these limits, the analysis must be discontinued, the cause determined, and the instrument recalibrated.

11.0 QUALITY CONTROL

11.1 Initial Calibration Verification (ICV). Analyzed immediately following calibration. The ICV is prepared from stock solutions from a standard source different than that of the calibration standard and described in the labnet data system. The ICV is analyzed at a concentration near the upper-point of the calibration curve. The results of the ICV are to agree within 10% of the expected value. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

11.2 Initial Calibration Blank (ICB). Analyzed immediately following the ICV. The ICB is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. The results of the ICB are to be one half the PQL. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

11.3 Interference Check Solutions (ICSA ,ICSAB). Analyzed at the beginning and end of each analytical run to verify the interelement and background correction factors. The ICSA and ICSAB are prepared from stock solutions as described in the labnet data system. Results are to be within 20% of the true value.

11.4 Continuing Calibration Verification (CCV). Analyzed prior to any samples, after every ten

samples, and at the end of an analytical run. The CCV is prepared from stock solutions as described in the labnet data system. The CCV is analyzed at a concentration near the mid-point of the calibration curve. The results of the CCV are to agree within 10% of the expected value. If not, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the previous ten samples.

11.5 Continuing Calibration Blank (CCB). Analyzed immediately following the CCV, after every ten samples, and at the end of an analytical run. The CCB is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. The results of the CCB are to be one half the PQL. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

11.6 Method Blank (PB). Analyzed along with the sample batch it was prepared with. The PB must be carried through the complete procedure and contain the same acid concentration as the samples used for analysis. The results of the PB are to be one half the PQL. If not, redigest all samples in the associated batch and reanalyze samples.

11.7 Laboratory Control Sample (LCS). Reagent water spiked with known quantities of all analytes carried through the complete procedure as the samples used for analysis. The results of the LCS are to agree within 20% of the expected value or vendor specified limits. The exception to this is silver and antimony, which are to agree within 50% of the expected value or vendor specified limits. If not, redigest all samples in the associated batch and reanalyze samples.

11.8 Matrix Duplicate. Analyzed at a frequency of one per matrix batch. A duplicate sample is brought through the entire sample preparation and analytical process in duplicate. A control limit of 20% RPD is used for sample values greater than ten times the instrument detection limit.

11.9 Matrix Spike and Spike Duplicate. Analyzed at a frequency of one per matrix batch. Sample spiked with known quantities of all analytes carried through the complete procedure as the samples used for analysis. A control limit of 25% of the actual value is used for sample values no greater than four times the spike added.

11.10 Post Digestion Spike (PDS). An analyte spike added to a portion of a prepared sample. A control limit of 25% of the known value determines if a matrix effect is suspected. The spike should produce a minimum of ten times and a maximum of one hundred times the instrument detection limit. The PDS should be analyzed at a frequency of one per matrix batch.

11.11 Low Level QC Check Standards (CRI and CRA). Analyzed at the beginning and end of an analytical run. The CRI and CRA are prepared from stock solutions as described in the labnet data system. The CRI is analyzed at a concentration near the PQL and the CRA is analyzed at

a concentration near the low-point of the calibration curve. The results of CRA are to agree within 50% of the expected value. The results of the CRI are to agree with 40% of the expected value.

11.12 Instrument Detection Limits (IDL). A minimum of seven replicate standards are analyzed at three to five times the expected IDL on three nonconsecutive days, and the average standard deviation multiplied by three to get the IDL. The IDL is analyzed quarterly.

11.13 Practical Quanitation Limits (PQL). The PQL is calculated as three to five times the MDL.

11.14 Method Detection Limit (MDL). A minimum of seven replicate standards are digested and analyzed following 40 CFR Part 136, Appendix B.

11.15 Linear Range is analyzed quarterly. Each individual analyte is analyzed near the upper limit of the range. The upper range limit is an observed signal no more than 10% below the level extrapolated from lower standards.

11.16 Interelement Correction (IEC). IEC's are analyzed with the primary interferences (Fe, Al, Cu, Ni, Mn, Cr, Co, V, Mo, Ti) being checked monthly or whenever maintainance is performed on the instrument. Upper range levels of each analyte are analyzed individually. Interelement correction equations are determined to compensate for the effects of interfering elements.

12.0 Procedure

12.1 Startup of the ICAP61

WARNING: Ensure that no pacemaker users are in the vicinity of the spectrometer. RF generator radiation may interfere with pacemaker operation.

A) Prior to initiation of the plasma, the operator should be familiar with the operator's manual supplied by Thermo Jarrell Ash.

B) Before starting the instrument, check the exhaust vents and cooling water to ensure they are operating.

C) Check that the pressure of the argon supply is 50 psi.

D) On the front of the instrument check that the standby (SB) and fatigue (FAT) lights are lit. At the back of the instrument check that the high voltage (HV) power switch is on. If the lights are not on, consult the Thermo Jarrell Ash operator's manual to conduct a "cold startup" (TJA 5.1.1).

E) Check that the drain tube is immersed in at least 8 inches of water in the plastic waste

container. Ensure there are no crimps in this tubing.

F) Start up is as follows:

1) Go to Setup on computer - hit [Enter].
2) Control F3 - hit [Enter] (This initiates the macro programed by TJA with all standard specifications.)

G) Connect the rinse and sample tubing on the peristaltic pump. Check that there is sufficient rinse solution in the rinse supply container. Start the pump, making sure that the sipper is immersed in the rinse solution.

12.2 Sample Evaluation

A) Locate the sample digestates to be analyzed and compare them to the applicable prep log in order to determine the analysis protocols. Determine which elements are required and prepare the appropriate calibration standards according to the standards preparation procedures.

B) Visually inspect the sample digestates, noting which samples were in duplicate and which were spiked. Also note initial and final sample volumes, which laboratory control samples were used and also the sample weights. Check this against the sample prep log for completeness for all these mentioned items. During this inspection any highly colored samples (usually an indication of high analyte concentrations) should be noted. These samples may require a rinse cup after them during analysis to minimize carryover effects.

C) Estimate the number of sample cups necessary for the autosampler table. Count also the possibility of all spiked samples needing a post-digest spike, as well as a serial dilution for each sample matrix.

12.3 Program Setup

A) On the main menu of the thermospec software cursor to the SETUP option and press ENTER.

B) The method name is selected at this time. The name can be either typed in or by pressing the <F6> function key to list all available programs.

C) The first page of the program will appear on the monitor. On the lower right hand portion of the screen the function keys will be listed with a variety of options. Select <F3> (SampInfo).

D) Cursor down 5 times to the default table names, specifically AUTOSAMPLER. Enter the name of the autosampler table you are about to create using the format: WMMDDYY. For instance, if today's date is February 6, 1999, then the autosampler table entered here would be: W020699.

E) Cursor to the default file names portion and enter under the data file the same name as the autosampler table, in this case: W020699.

F) Press the function key <F9> (done/keep) until back to the setup option (entered twice). The program now has stored the name of the new autosampler table and the name of the new data file in which the information is to be stored.

12.4 Setup of the Autosampler Table

A) On the main menu cursor once again to the setup mode and press enter.

B) Cursor through the setup options to AUTOSAMPLER and press enter.

C) As with the program portion, here you will be required to enter an autosampler table name. Use the name selected in the program portion, which, in this case is: W020699 and press enter.

D) The autosampler screen will display information regarding this specific table. At this point under "set # description", it will read, "this table is empty". Choose the <F3> (add set) function key to enter the information for this table.

E) For all analyses use the following entries here:

"SET DESCRIPTION" - leave blank
"METHOD NAME" - current method in use
"DEFAULT COMMENT" - enter the SDG number
"DEFAULT OPERATION ID" - enter the initial of the ICAP operator
"LABORATORY ID" - enter the initials of the person who prepared the samples
"CUSTOMER ID" - enter the date that the samples were prepared using MM/DD/YY format
"AUTO-RINSE BETWEEN SAMPLES?" - leave YES
"RINSE TIME" - enter 50 seconds
"# SAMPLE POSITIONS NEEDED" - enter the number of cups required.

Cursor down to "default limit check table name" - and type in LIMITS.

This page is complete. Now press <F1> (ed samples) to set up the sample cup designations.

F) Press <F5> (ins stds). This allows the insertion for a standardization standard.

G) Initial QC must now be entered. Press <F3> (insQC) and type ICV1 then press enter. Cursor to the check tables column and type in again ICV1 and press enter. Cursor back to the next sample name column and press <F3> again. This time type ICB. Use this same procedure to enter:

<F3> ICV1----------ICV1
<F3> ICB----------ICB
<F3> CRII----------CRII
<F3> ICSAI---------ICSAI
<F3> ICSABI--------ICSABI
<F3> CCV1----------CCV1
<F3> CCB1----------CCB1

All initial QC is now entered.

H) To enter the sample names, a specific format must be utilized for in order to facilitate proper data storage in the appropriate software files.

I) QC must be analysed after every 10 samples. In the case of initial QC, the CRI, ICSA and ICSAB count as samples. Therefore, only 7 actual samples can be analyzed before the next series of QC.

J) The final QC is run in the following order: CRIF, ICSAF, ICSABF, CCV_, and CCB_.

K) Double check autosample table making sure all samples and QC are entered correctly.

L) In order to store the completed autosampler table press <F9> (done/keep), then press again to get back to the main menu.

M) Cursor through setup and go to the autosampler section again to check that the table is in memory. Check paper supply to be sure there is enough for the entire run. Then press <F2> to print out the table. Press <F9> (done/keep) to get back to the main menu.

N) Using the printed autosampler table, pour fresh standards and all the samples into the cups and test tubes and place them in their designated positions on the autosampler tray.

13.0 ANALYSIS

13.1 Cursor to the main menu. Select the operation mode and press enter. Select ANALYSIS and press enter. Once again the method name will be requested. Type the current method and press enter. To initiate the analysis, press <F9> (autosampler). This will call the autosampler table from memory. On the monitor should be the first sample, usually STD1. Check that the sipper is firmly in the autosampler, and the height is adequate for both standard cup and test tube levels. To start the analysis, press <F1> (run). The autosampler will move to the first sample and begin operation.

14.0 CALCULATIONS

14.1 Calculations are performed by the analytical software with direct concentration readout. The conversion to mg/Kg is as follows:

$$\frac{\text{(Reading in ppb)(sample digestate volume in liters)}}{\text{Sample Wt.in grams X dec. \% solids}} = \text{results in mg/Kg}$$

14.2 All data is downloaded to the labnet data system.

A. In the thermospec software go to IMS, then to FILER, then to Samples Archival. Enter the date of analysis (the time is not necessary). Press F1 (New Path) and type C:\ and the name of run being downloaded with the suffix .ARC. Enter. Press F9 (done/go). Escape to exit.
B. Exit out of the thermospec software to windows. Enter windows explorer.
C. Open C: find the data to be downloaded. Drag the file to L: directory.
D. Open L: find the data to be downloaded and drag file to either ICAP1 or ICAP2 depending on which instrument the data is from.

15.0 ACCEPTANCE CRITERIA

Acceptance criteria can be found in Quality Control Section 11 for all QC samples.

16.0 POLLUTION PREVENTION

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of Chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 WASTE MANAGEMENT

17.1 All waste shall be managed in accordance with all state and federal requirements. See the STL-CT Hazardous Waste Management Plan.

17.2 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

18.0 SUPPLEMENTAL DOCUMENTS - None

19.0 REFERENCES

18.1 USEPA SW846 Third edition Method No. 6010B.

20.0 SUBSTANTIVE REVISIONS

20.1 Original issue - 01/25-95
20.2 Changed name on SOP, reformatted to meet QA requirements; 1/20/03.

Table #1

Quality Control Sample	Control Limit	Failure Action
ICV	± 10 %	Recalibrate
ICB	± ½PQL	Recalibrate
CRI	± 20 %	Recalibrate
ICSA	± 20 %	Recalibrate
ICSAB	± 20 %	Rerun Samples
CCV	± 10 %	Rerun Samples
CCB	± ½PQL	Rerun Samples
Duplicate	± 20 % RPD	Flag Sample
Sample Spike	± 25 % Recovery	Flag Sample
Prep Blank	± PQL	Reprep Samples
Lab Control Sample	± 20 %, 95 % Confid Win	Reprep Samples

Severn Trent Laboratories - Connecticut

SOP for ION CHROMOTOGRAPHY – Method CVS06100.CT
Revision 0
Date Effective: 04/25/2000

This documentation has been prepared by Severn Trent Laboratories (STL) solely for STL's own use and the use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to Severn Trent Laboratories upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF SEVERN TRENT LABORATORIES IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY SEVERN TRENT LABORATORIES IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2000 SEVERN TRENT LABORATORIES, INC. ALL RIGHTS RESERVED.

This is a controlled Document if Stamped in Red:

_____

Issued to: _____

1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in the SOP for SOP's.

Laboratory Director

Quality Assurance Manager

Group Leader

2.0 SCOPE AND APPLICATION

2.1 The method covers the determination of the following inorganic anions:

Bromide	Nitrite-N
Chloride	Ortho-Phosphate-P
Fluoride	Sulfate
Nitrate-N	

2.2 The matrices applicable to this method are as follows:
Drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (using the extraction procedure given in section 8.5), and leachates (when no acetic acid is used).

2.3 The range tested for each anion is as follows:

Analyte	Range, mg/L
fluoride, bromide, nitrite-N, nitrate-N, O-phosphate-P	0.1 to 5
chloride	0.5 to 50
sulfate	1 to 100

Note. The calibrated range for this method is two orders of magnitude. These ranges were based on a 50 uL sample loop.

2.4 When this method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a fortified sample matrix covering the anions of interest.

2.5 Bromide and nitrite react with most oxidants employed as disinfectants for drinking

waters. The utility of measuring these anions in treated water should be considered prior to conducting the analysis.

2.6 The document control number for this SOP is CVS06100.CT.

3.0 TERMS AND DEFINITIONS

3.1 Refer to the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 A small volume of sample is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, an analytical column, a suppressor cartridge, and a conductivity detector.

4.2 An extraction procedure must be performed to use this method for solids (See Section 12.8).

4.3 The sample is injected into a stream of carbonate-bicarbonate eluent that carries it through three different ion exchange columns and into a conductivity detector.

4.4 The first two columns, a guard column and a separator column, are packed with low-capacity, strongly basic anion exchanger. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin.

4.5 The last column is a suppressor column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions into their respective acids.

4.6 The separated anions in their respective acid forms are then measured by the conductivity detector and quantified using a calibration curve generated from known standards.

4.7 The anions are identified by their retention times compared to known standards.

4.8 This method is based on EPA Method 300.0 and SW846 method 9056.

5.0 INTERFERENCES

5.1 Interferences can be caused by substances with retention times that are similar to and overlapping those of the analyte of interest. Large amounts of an analyte can interfere with the peak resolution of a closely eluting analyte. Sample dilution and/or fortification can be used to solve many interference problems associated with overlapping retention times. However, sample dilution may typically affect the MDL values proportionally.

5.2 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.

5.3 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 small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant. It is, however, the responsibility of the user to generate precision and accuracy information in each sample matrix.

5.4 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 adjustments.

5.5 The quantitation of unretained peaks should be avoided, such as low molecular weight organic acids (formate, acetate, propionate etc.) which are conductive and coelute with or near fluoride and would bias the fluoride quantitation in some drinking waters and most wastewaters.

6.0 SAFETY

6.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable.

6.2 At a minimum, wear PVC gloves, a lab coat, and eye protection when handling samples and reagent. Use a suitable fume hood for the analysis.

6.3 Always refer to your STL Employee Safety Handbook prior to performing laboratory operations.

7.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

7.1 Samples are collected in plastic or glass bottles. All bottles must be used only once. Volume collected should be sufficient to ensure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

7.2 Sample preservation and holding times for the anions that can be determined by this method are as follows:

Analyte	Preservation	Holding time
Bromide	cool to 4^0C	28 days
Chloride	cool to 4^0C	28 days
Fluoride	cool to 4^0C	28 days
Nitrate-N	cool to 4^0C	48 hours
Nitrite -N	cool to 4^0C	48 hours
Nitrate/Nitrite (Combined)	conc. H_2S0_4 to pH <2	28 days
Ortho phosphate-P	cool to 4^0C	48 hours
Sulfate	cool to 4^0C	28 days

8.0 EQUIPMENT AND SUPPLIES

8.1 Balance -- analytical, capable of accurately weighing to the nearest 0.0001 g.

8.2 Glassware--Class A volumetric flasks and pipettes as required.

8.3 Ion Chromatograph -- Analytical system complete with ion chromatograph and all required accessories including analytical columns and detectors.

8.3.1 Anion guard column: QS-AlG (Lachat part no.28085) A protector of the separator column. If omitted from the system, the retention times will be shorter.

8.3.2 Anion profiling column: QS-A1 (Lachat part no.28084).

8.3.3 Anion suppressor cartridge: QE-Al (Lachat part no.28097). The replacement suppressor cartridge has Lachat part no 28098.

8.3.4 Detector -- Temperature controlled conductivity detector: CM- 100. Approximately

0.25 µL internal volume.

8.4 Lachat's Omnion IC Software.

9.0 REAGENTS AND STANDARDS

9.1 Sample bottles: glass or polypropylene of sufficient volume to allow replicate analyses of anions of interest.

9.2 Reagent water: Use ASTM Type II water (nanopure water) for all solutions.

9.3 **Eluent solution: Prepare Daily. 2.0 mM Sodium bicarbonate** (CASRN 144-55-8), **2.6 mM sodium carbonate** (CASRN 497-19-8). Dissolve **0.1680 g sodium bicarbonate** ($NaHCO_3$) and **0.2755 g of sodium carbonate** (Na_2CO_3) in reagent water (9.2) and dilute to **1 L**. Degas the eluent by helium sparging (one minute for each liter) or by vacuum sonication. Filter through a 0.22 µm nylon filter.

It has been observed that phosphate and sulfate peaks are not resolved when the eluent is prepared using oven-dried and desiccated chemicals. Therefore, to prepare the eluent, use chemicals directly from the vendor's bottle.

9.4 **Regenerant**: Sulfuric acid (CAS RN 7664-93-9) 0.25 M

Regenerant Stock Solution, 1.0 M sulfuric acid

To a 1L volumetric flask containing 800 ml of reagent water add 56 ml of concentrated sulfuric acid (H_2S0_4). Caution the flask will become warm. Cool to room temperature and dilute to the mark. May be stored at least 4 weeks.

Working Regenerant, 0.25 M sulfuric acid

In a 1 L container, add 250 mL of Regenerant Stock Solution and 750 mL reagent water. Stir to mix. May be stored at least 4 weeks.

Alternative: To 1L volumetric containing 800 ml of reagent water, add 14 ml of concentrated sulfuric acid and dilute to volume with reagent water. Mix by inverting three times.

9.5 20 % Methanol (self-flush reagent for eluent pump)

To a 200 ml container, add 40 ml methanol (CH_3OH). Fill with reagent water and invert to mix.

9.6 **Stock Standard Solutions, 1000 mg/L (1 mg/ml):** 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. To a 1.0 L volumetric flask, add the exact weight of reagent listed below and fill the flask to the mark with DI water. Stir to dissolve.

REAGENT	Weight (g)	Concentration
Sodium Fluoride NaF	2.2100	1000 mg F/L
Sodium Chloride NaCI	1.6485	1000 mg Cl/L
Sodium Nitrite $NaNO_2$	4.9257	1000 mg NO_2-N/L
Sodium Bromide NaBr	1.2876	1000 mg Br/L
Sodium Nitrate $NaNO_3$	6.0679	1000 mg NO_3-N/L
Potassium Phosphate KH_2P04	4.3937	1000 mg HPO_4-P/L
Potassium Sulfate K_2SO_4	1.8141	1000 mg SO_4/L

9.7 **Diluted Individual Stock Standard Solutions, 100 mg/L (0.1 mg/ml):**

To separate 250 ml volumetric flasks or containers, add 25 ml of the indicated Stock Standard Solution (7.6). Fill to the mark with reagent water. Stir to mix.

Stock Standard:(9.6) 1000 mg/L	Number of ml	Final Number	Concentration mg/L
Fluoride, Chloride, Nitrate-N, Bromide, Nitrate-N, Phosphate-P Sulfate	25	250	100 mg/L

NOTE: Stability of standards: Stock Standard Solutions (9.6) are stable for at least one month when stored at 4°C. Diluted Stock Standard Solutions (9.7) are prepared weekly and stored 4°C. Standards that contain nitrite and phosphate are prepared fresh daily.

9.8 Working Mixed Standard A

To a 200 mL volumetric flask or container, transfer the volumes of the Stock Standard Solutions (9.6) or the Diluted Individual Stock Standard Solutions (9.7) listed below. Fill to the mark with DI water. Invert to mix. Store at 4°C.

Stock Standard	Number of ml	Concentration mg/L
Fluoride 100 mg/L	10	5
Chloride 1000 mg/L	10	50
Nitrite-N 100 mg/L	10	5
Bromide 100 mg/L	10	5
Nitrate-N 100 mg/L	10	5
Phosphate-P 100 mg/L	10	5
Sulfate 1000mg /L	20	100

9.9 **Working Mixed Standards B through F**

Working Mixed Standards B through F are prepared by diluting Working Mixed Standard A (9.8) as listed below. Standard A is repeated for reference.

Std	Standard A ml	Final sol'n ml	Concentration (mg/L)						
			F^-	Cl-	NO_2^--N	Br-	NO_3^--N	HPO_4^{2-}	SO_4^{2-}
A	----	----	5.0	50.0	5.0	5.0	5.0	5.0	100.0
B	30.0	50	3.0	30.0	3.0	3.0	3.0	3.0	60.0
C	20.0	100	1.0	10.0	1.0	1.0	1.0	1.0	20.0
D	20.0	250	0.4	4.0	0.4	0.4	0.4	0.4	8.0
E	10.0	500	0.10	1.00	0.10	0.10	0.10	0.10	2.0
F	10.0	1000	0.05	0.5	0.05	0.05	0.05	0.05	1.0

Example: To prepare standard B, add 30 ml of standard A to a 50-mL volumetric flask or bottle. Fill to the mark.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Prepare reagents as described in Section 9. Calibration standards are prepared at six concentration levels, excluding the blank. The recipe for the calibration standards is in Section 9. The calibration standards employed must bracket the concentration of the analytes of interest in the unknown samples.

10.2 Set up the IC manifold according to the flow diagram as shown in the appendix section.

Inspect carefully for proper connections.

10.3 Input the data system parameters as shown in the appendix section.

10.4 Calibrate the instrument, following the start-up procedures given in Section 11. First run an injection of DI water or a standard to completion. Then run the calibration standards in succession. After calibration, a blank should be run for validation. The calibration criterion is a correlation coefficient of ≥ 0.995.

10.5 The data system will associate the known concentrations with the peak heights or peak areas recorded for each standard. These responses are then used to prepare a calibration curve for each analyte.

10.6 The calibration must be verified on each working day with an ICV, or whenever the eluent is changed, and after every 10 samples with a CCV. If the response 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% off the expected values, a new calibration curve must be prepared for that analyte.

10.7 Nonlinear response can result when the analytical column capacity is exceeded (overloading). The responses for the sample when diluted 1:1, and when not diluted, should be compared. If the determined results are the same, accounting for dilution, then the samples having this total ionic concentration need not be diluted.

11.0 QUALITY CONTROL

11.1 Initial Calibration Verification (ICV)

Immediately after the Lachat Ion Chromatograph Analyzer has been calibrated, the accuracy of the initial calibration shall be verified and documented by the analysis of the Initial Calibration Verification Standard. The criterion of the ICV is ± 10 % of true value.

11.2 Continuing Calibration Verification (CCV) {Instrument Performance Check Solution}

To ensure calibration accuracy during each run, a mid-range standard must be used for continuing calibration verification and must be analyzed at a frequency of 10% of samples. The standard must be analyzed at the beginning of the run, after ten samples, and after the last analytical sample. The criteria for CCV is ± 10% of the true value, and the preceding CCV should be ± 5% of the first CCV.

11.3 Laboratory Control Samples (LCS) { Quality Control Samples}

To ensure calibration accuracy during each run, an LCS must be run with every batch of twenty samples. The LCS is obtained from a commercial supplier i.e. Analytical Products Group. The criterion for the LCS is ± 15 % of the true value.

11.4 Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses

A calibration blank must be analyzed at the beginning of the run, after every initial and continuing calibration verification, at a frequency of 10 percent of samples and after the last analytical sample.

At least one preparation blank consisting of reagent water must be analyzed with each batch of twenty or fewer samples run.

The values for the preparation blank must be recorded in mg/L for aqueous samples and in mg/Kg for solid samples.

11.5 Spike Sample and Spike Blank Analysis

One blank spike must be performed with each batch of samples. The blank spike data will be used to assess the laboratory performance against the required control limits of ± 10% of the true value.
The spike sample analysis is designed to provide information about the effect of the sample matrix on the measurement methodology. At least one spike must be performed on each group of twenty or fewer samples of a similar matrix (i.e. water, soil). Samples identified as field blanks cannot be used for spike sample analysis. Control limit for the matrix spike recovery is ± 20 %.

11.6 Duplicate Sample Analysis

One duplicate sample shall be analyzed from each group of ten or fewer samples of a similar matrix type (i.e. water, soil). Samples identified as field blanks cannot be used for duplicate analysis. The criterion for duplicate analyses is an RPD ≤ 20 %.

11.7 Method Detection Limit (MDL) Determination

Annually, the method detection limit (in mg/L) shall be determined by multiplying by 3.14,

the average of the standard deviation obtained from the analysis of seven digested standard solution replicates at a concentration up to five times the estimated Instrument Detection Level.

11.8 Instrument Maintenance

An instrument maintenance log is kept for the Lachat Autoanalyzer.

11.9 Labeling and Coding

All standards, reagents, and LCS's are labeled and coded according to the Classical Chemistry SOP for Labeling and Coding of Standards.

12.0 PROCEDURE

12.1 Follow the sample collection, pretreatment and preservation procedures given in Section 7.

12.2 Turn on the power to all modules.

12.3 Add eluent to the eluent reservoir, and prime the eluent delivery pump. Be sure that all air bubbles are eliminated from the eluent inlet filter and the eluent inlet tubing. Tap the inlet filter on the bottom of the eluent container to dislodge bubbles. To rewet a brand-new or dried-out inlet filter, unscrew the filter from the inlet line, push methanol backward through the filter using a wash bottle or syringe. Rinse thoroughly with DI water, and reconnect the filter to the inlet line.

12.4. With the columns disconnected, pump eluent until the connecting tubing is flushed and all air is removed. Stop the pump and then connect the columns. Briefly turn on the eluent delivery pump to check for leaks and excessive back pressure.

12.5. Add DI water and regenerant to the respective reservoirs, as appropriate to the method.

12.6. Initialize the instrument.

12.6.1. For QC 8000: With the regenerant and DI wash lines in DI water, turn on the peristaltic pump and check for leaks around the suppressor. Then place these lines into the respective containers.

12.7. Place samples (which have been filtered through a 0.22 μm nylon filter) in the autosampler. Input the sample identification, run order, vial number, and number of replicates as required by the data system and the QC procedure.

12.8 Solid Material Extraction Procedure:

Add an amount of reagent water ten times the weight of solid material taken as sample. Mix the slurry for ten minutes using a magnetic stirring device. Filter the slurry through a 0.22 μm nylon filter. Care should be taken to make sure that identification of peaks and resolution of the peaks is good. A solid spike should be run with the soil samples. Spiking should be done prior to sample extraction.

12.9 Start the eluent pump and the peristaltic pump. Submit the Batch for analysis.

NOTE : ***An initial injection of DI water or a standard should be run as the first sample.***

In order to perform USEPA compliance monitoring, the suppressor cartridge must be in-Line during sample processing. Any data generated without the in-Line, fully activated suppressor cartridge, cannot be reported for compliance monitoring.

12.10. If one or more analyte responses exceed the calibration range, dilute the sample with reagent water or an appropriate diluent and reanalyze.

12.11. Should more complete resolution be needed between peaks, the eluent can be diluted 10% to 30%. This will increase separation but will also cause the later eluting analytes to be retained longer. This dilution should not be considered a deviation from the method. Alternately, slowing the eluent flow rate by 20% to 40% can increase separation slightly.

12.12. System shut-down. At the end of each day's work, flush all columns with DI water. If the system will be idle for longer than three days, disconnect the columns and suppressor and install end caps. Refer to the information sheet provided with the analytical column for specific information relating to column care, including long-term storage and column cleaning procedures. To flush the regenerant, place the regenerant inlet line in DI water. Pump reagent water through the system. For long-term storage of the eluent pump, prime with isopropanol.

12.13. SYSTEM NOTES

12.13.1. For information on system maintenance and troubleshooting refer to the System Operation Manual and Trouble Shooting Guide.

12.13.2. Do not suddenly release the pressure from the columns or they may be damaged.

12.13.3. The retention times for each of the analytes are affected by eluent concentration, flow rate, extreme range of analyte concentration, and column performance. The user may need to make minor adjustments in the Integration Events Table and the Peak Table to ensure each peak is identified correctly and integrated properly.

12.13.4 As preventative maintenance, the guard column should be replaced periodically.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Calibration is done by injecting standards. Analyte concentrations for unknown samples are calculated from the respective regression equations. The calibration criterion is a correlation coefficient of ≥ 0.995.

13.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest calibration standard should be diluted and reanalyzed. Sample concentrations less than the lowest calibration standard should not be reported unless the MDL standard is included in the calibration model.

13.3 Report results in mg/L.

13.4 Report NO_2 as N
NO_3 as N
HPO_4^{2-} as P

13.5 Concentration in Samples:

13.5.1 Calculate the aqueous samples in mg/L of original sample, as follows:

mg /L = A x D If dilution factor is not entered in the Lachat IC tray.

where: A = mg /L sample from Lachat print-out.

D = any other dilution factor necessary for sample to be below the high standard.

The minimum value that can be substituted for A is the practical quantitation limit.

13.5.2 Calculation of solid samples in mg/Kg of original sample, as follows: (A separate determination of percent solids must be performed: Appendix II.)

The concentration of sample is determined as follows:

$$mg/Kg = \frac{A \times D \times F}{B \times E}$$

where: A = mg /L of sample from Lachat print-out
B = wet weight of original sample **(in g)**.
D = any other dilution factor necessary for sample to be below the high standard.
E = % solids (See Appendix II)/100
F = final sample volume **(in ml)** after extraction.

The minimum value that can be substituted for A is the practical quantitation limit.

13.6 Percent Recovery

13.6.1 Spike recoveries are calculated as follows:

$$\% \text{ Recovery} = \frac{(SSR - SR) \times 100}{SA}$$

where: SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

When sample concentration is less than the practical quantitation limit, use SR = 0 for calculating percent recovery.

13.6.2 Percent Recovery of Initial, Continuing Calibration Verification and LCS

For Initial Calibration Verification (ICV):

$$\% \text{ Recovery} = \frac{\text{Found (ICV) value} \times 100}{\text{True (ICV) value}}$$

For Continuing Calibration Verification (CCV):

$$\% \text{ Recovery} = \frac{\text{Found (CCV) value} \times 100}{\text{True (CCV) value}}$$

For Laboratory Control Sample (LCS):

$$\% \text{ Recovery} = \frac{\text{Found LCS value} \times 100}{\text{True LCS value}}$$

13.7 Relative Percent Difference

The Relative Percent Difference is calculated as follows:

$$RPD = \frac{(S - D) \times 100}{(S + D)/2}$$

where: S = Sample result and D = Duplicate result

14.0 ACCEPTANCE OF DATA

14.1 Initial Calibration Verification (ICV): The control limit for the ICV is ±10 % of the true value.

Action on failure: When measurements exceed the control limit, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration verified.

14.2 Continuing Calibration Verification (CCV) : The control limit for the CCV is ±10% of true value, and the preceding CCV should be ± 5% of the first CCV.

Action on failure: If the deviation of the CCV is greater than the control limit, the preceding ten samples analyzed since the last compliant calibration verification must be reanalyzed. If recalibration is necessary, an ICV must be run to verify the calibration.

14.3 Blank Spike (BS): The control limit for the BS is ±10 % of true value.

Action on failure: If the deviation of the BS is greater than the control limit, the BS must be reanalyzed. If recalibration is necessary, an ICV must be run to verify the calibration.

14.4 Initial Calibration Blank (ICB), and Continuing Calibrations Blank (CCB): The absolute value of the concentration in the blank must be less than the PQL.

Action on failure: If the absolute value of the calibration blank result exceeds the PQL, reanalyze the preceding ten analytical samples or all analytical samples analyzed since the last compliant calibration blank. If recalibration is necessary, an ICV must be run to verify the calibration.

14.5 All preparation blank results must be less than the PQL.

Action on failure: If the concentration in the blank is above the PQL, the lowest concentration of sample in the associated samples must be ≥ 10X the blank concentration. Otherwise, all samples associated with the blank with a concentration less than 10X the blank concentration and above the PQL must be reanalyzed with a new blank that meets criteria.

14.6 Spike Sample Recovery: The acceptable range for spike recovery is ± 20 % percent of the spike amount added; no spike criteria is applied if the sample concentration exceeds the spike level by a factor of four or more.

Action on failure: When the spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added, the spike must be rerun. If it still fails criteria and all other QC and blank spike passes then the cause of the failure is matrix related.

14.7 Duplicate Analysis RPD: The acceptable RPD is less than or equal to 20%.

Action on Failure: Another duplicate has to be analyzed.

15.0 REPORTING OF RESULTS

15.1 Results are reported by entering the data collected into SEEDPAK.

15.2 Batch quality control results are checked and reported.

15.3 Case narratives should include information received on corrective action reports, any holding time problems, and any other problems associated with sample analysis.

16.0 POLLUTION PREVENTION

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of

wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 WASTE MANAGEMENT

17.1 All waste shall be managed in accordance with all state and federal requirements. See the STL-CT Hazardous Waste Management Plan.

17.2 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 SOP for Terms and Definitions.

18.2 Classical Chemistry SOP for Labeling and Coding of Standards.

19.0 REFERENCES

19.1. Pfaff, S.D., USEPA Method 300.0, "Determination of Inorganic Anions by Ion Chromatography". EMSL, Office of Research and Development, USEPA, Cincinnati,

OH 45268, USA, rev. 2.1, 1993.

19.2 SW846 method 9056, "Determination of Inorganic Anions by Ion Chromatography". Revision 0, September 1994.

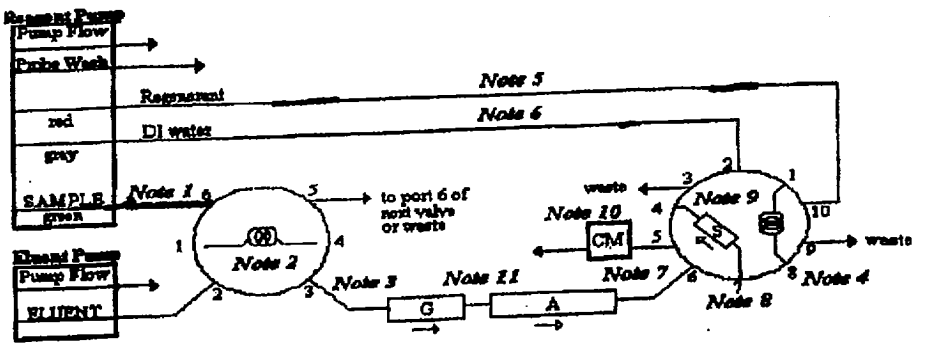
19.3 Lachat IC Operating Manuals, Lachat Instruments Inc., Milwaukee, WI, USA.

20.0 REVISIONS

20.1 Original Document.

APPENDIX

MANIFOLD DIAGRAM FOR QC 8000(12-16 Channel Peristaltic Pump)



C	guard column QS-A1G (Lachat part no 28085)
A	analytical column QS-A1 (Lachat part no.28084)
S	suppressor cartridge QE-Al Lachat part no.28097) *The suppressor cartridge* is *replaceable (replacement cartridge part no. 28098)*
C M	conductivity module

Note#	Tubing Type	Connected to:
*Note 1	Green/Green sample line	six-port valve port 6
Note 2	PEEK sample Loop – 200 µL	six-port valve port1 to port 4
Note 3	Yellow PEEK Tubing - 25 cm	six-port valve port 3 to guard column
Note 4	0.8 mm id Teflon Tubing - 225cm	ten-port valve port 1 to on 8
** Note 5	Red/Red sulfuric acid line	Peristaltic pump to ten-port valve port 10
***Note 6	Gray/Gray DI water line	Peristaltic pump to ten-port valve port 2
Note 7	Yellow PEEK Tubing - 34 cm	analytical column to ten-port valve port 6
Note 8	Tan PEEK Tubing - 12 cm	ten-port valve port 7 to Suppressor
Note 9	Tan PEEK Tubing - 12 cm	suppressor to ten-port valve port 4
Note 10	Yellow PEEK Tubing - 20 cm	CM inlet to ten-port valve port 5
Note 11	Yellow PEEK Tubing - 6 cm	guard to analytical column

* ***Green/Green sample tube can be replaced with RedlRed to reduce sample consumption if running IC alone without dilutor. Use Green/Green tubing if running IC with FIA or IC with dilutor.***

** ***Connect Red/Red pump tubing to port 10 (ten-port valve) using 100 cm of 0.8mm i.d.***

Teflon tubing.

* * * *Conned Gray/Gray pump tubing to port 2 (ten-port valve) using 100 cm of 0.8 mm id Teflon tubing.*

PARAMETERS AND VALIDATION DATA

Parameters

Separation Conditions

Eluent:	2.0 mM $NaHCO_3$ + 2.6 mM Na_2CO_3
Regenerant:	0.25 M sulfuric acid
Sample loop:	100 µL
Expected pressure:	1250 ± 250 psi

Pump Settings

Eluent flow rate:	2.0 ml/min
High pressure limit:	1500 psi
Low pressure limit:	500 psi

Acquisition Setup

Sample Frequency:	1Hz
Run Time*:	12 ± 1 min
Channel Status:	On
Analyze After Acq.:	On
Trigger Type:	Contact open
Acquisition Delay:	1.4 mm
Probe in Sample:	65 s
(Set under Method Menu)	

* ***Run Time*** *is the time required to return to baseline after the last peak is eluted. The test sheet included with the analytical column shows the actual Run Time for that column.*

Integration Events Table A*

Event	Start Time	Stop Time	Value
Integration Off	0	*1.7*	0
Threshold	1.5	1.9	750
Width	2.2	2.5	0.3
Valley to Valley	2.3	5.5	0
Shoulder Sensitivity	2.3	3.1	100
Threshold	3.5	4.5	400
Width	4.5	5.1	0.6
Valley to Valley	8.0	9.0	0
Width	7.0	8.0	1.1

** Sample table only. The experienced user may program these events graphically.*

Peak Table

Name	Retention Time*	Window*	Units
Fluoride	2.3	0.3	mg/L
Chloride	3.25	0.5	mg/L
Nitrite-N	3.9	0.5	mg/L
Bromide	5.25	0.7	mg/L
Nitrate-N	6.2	0.9	mg/L
Phosphate-P	8.25	1.1	mg/L
Sulfate	10.0	2.0	mg/L

**These parameters may have to be adjusted for the software to label each analyte peak correctly.*

Calibration Setup

Time Unit	min
*Peak Attribute/Quantitate	**
*Calibration Fit/Fit Type	linear
*Weighting Method	1/x
*Force Through Zero	no
*Peak Area/Calib. Flag	replace
*Scaling	None
*RT Update After Calib.	No
*RT Update After Run	No
ISTD Amount	0
Sample Amount	1
Multiplication Factor/Manual Dilution	1

** In Omnion IC version 2.0 these parameters are selected in the Peak table. Version 2.0 also allows differing parameters for each analyte.*

** Use either Peak heights or Area for the anions.

External Events Table

B. External Events Table for QC8000 IC+ Automated Ion Analyzer

Event Name	Time	1	2	3
LOAD	0.05	0	0	1
INJECT	1.4	0	0	0

This example assumes that the IC SPM cable is connected to VALVE 3 on the valve distribution panel. The LOAD event actuates VALVE 3 as per column 3 of the table.

Display **Options - Channel A**

X-axis min	0 min
X-axis max.	12 mm
Y-axis min	-25 µS/cm
Y-axis max.	350 µS/cm
Attenuation:	1024

Detector Configuration*

Type:	Analog
Name:	Conductivity
Connected to Analog Input #:	**
Y-Axis Label:	µS/cm
Y-Axis Multiplier:	0.0001
Maximum # of Peaks:	50
Maximum # of Named Peaks:	25

** *For QC 8000 select the letter that indicates the channel used by IC on the detector distribution board.*

Detector Settings

Zero/Background:	Background
Gain:	1
Fast Response:	off
Temperature Setting:	33°C
Inverse Polarity:	off

PERCENT SOLIDS DETERMINATION PROCEDURE

1. Weigh out 5-10 g of sample to a tared weighing dish. Weigh and record the weight to the nearest 0.01 g.

2. Place weighing dish plus sample in a drying oven maintained at 103-105^{0}C. Sample handling and drying should be conducted in a well-ventilated area.

3. Dry the sample overnight (12-24 hours) but no longer than 24 hours. If dried less than 12 hours, it must be documented that constant weight was attained. Remove the sample from the oven and cool in a dessicator with the weighing dish cover in place before weighing. Weigh and record weight to nearest 0.01 g. Do not analyze the dried sample.

NOTE: Drying time is defined as the elapsed time in the oven; thus raw data must record time in and out of the oven to document the 12 hour drying time minimum. In the event it is necessary to demonstrate the attainment of constant weight, data must be recorded for a minimum of two repetitive weigh/dry/dessicate/weigh cycles with a minimum of 1 hour drying time in each cycle. Constant weight would be defined as a loss in weight of no greater than 0.01 g between the start weight and final reweigh of the dried sample.

4. Duplicate percent solids determinations are required at a frequency of one in every twenty samples or less.

5. For the duplicate percent solids determination, designate one sample aliquot as the "original" sample and the other aliquot as the "duplicate" sample. Calculate dry weight using the results of the "original" sample aliquot.

6. Calculate percent solids by the formula below. This value will be used for calculating analytical concentration on a dry weight basis.

$$\% \text{ Solids} = \frac{\text{Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

7. Calculate the RPD of the % solids as follows:

$$RPD = \frac{(S - D) \times 100}{(S + D)/2}$$

where: S = Sample result
and D = Duplicate result

The acceptable RPD is 20 %.
Action on Failure: Another percent solid has to be determined.