QUALITY ASSURANCE PROJECT PLAN
FORMER BARKSDALE FACILITY
BARKSDALE, WISCONSIN

Date: September 6, 2001

Project No: 7354

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CORPORATE REMEDIATION GROUP
An Alliance between
DuPont and URS Diamond

Barley Mill Plaza, Building 27
Wilmington, Delaware 19805

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Site Name: Former Barksdale Facility

Site Location: Barksdale, WI

Document Title: Quality Assurance Project Plan

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Lead Organization: DuPont Corporate Remediation Group

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QUALITY ASSURANCE PROJECT PLAN

FORMER BARKSDALE FACILITY

BARKSDALE, WI

Project No. D4BA7354.01

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PROJECT PERSONNEL SIGN-OFF SHEET

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1.0 INTRODUCTION

The former Barksdale Works Grounds (see Figure 1) occupies approximately 1800 acres along State Highway 13 and the shore of Lake Superior between Ashland and Washburn, Wisconsin. DuPont operated the plant from 1904 to 1971, primarily for the manufacture of explosives. Subsequent to the sale of the property in 1986, preliminary characterization of the soil and groundwater conditions at the site were initiated in 1997 (Site Conditions Report, Dupont, 1997). Since that time, additional screening of surface soils, the installation of monitor wells on the property, and the periodic monitoring of residential wells in the vicinity of the property has been conducted. The original Quality Assurance Project Plan (QAPP) for this site was developed by DuPont in October,1997, in conjunction with the preliminary site investigation work.

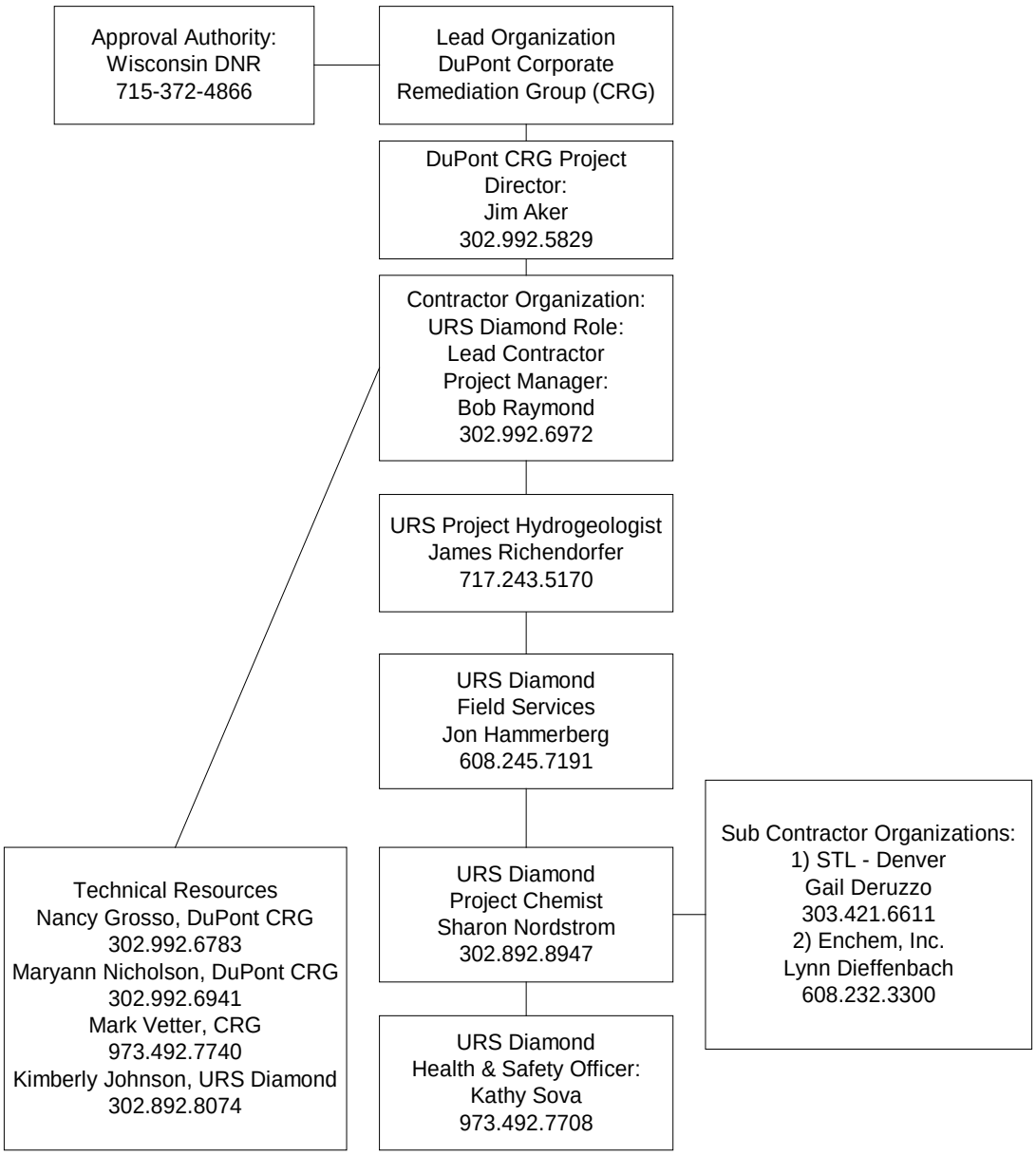
The primary purpose of this document is to update and expand the sampling, analytical, and quality assurance requirements established in the original QAPP in support of additional site characterization and groundwater monitoring activities proposed for 2001-2002.

This QAPP is a dynamic document designed to be amended, as necessary, to address all site groundwater, surface water, soil, and sediment sampling events. All future sampling will be developed in accordance with this QAPP. However, if the QAPP does not provide the specific sampling, analytical, and quality assurance procedures necessary to implement future activities, the QAPP will be updated, or a QAPP addendum will be developed to provide the appropriate procedures.

1.1 Project Organization

The project team is composed of an interdisciplinary team of DuPont, URS Diamond, and subcontractor personnel. The figure below summarizes the key individuals, respective role/responsibility as currently identified, and organization. A brief description of key management staff responsibilities is provided in Section 1.3.

ORGANIZATIONAL CHART



1.2 Key Management Staff

1.2.1 Project Director

Mr. James Aker, DuPont CRG, will serve as the Project Director for this project. Mr. Aker has the responsibility for the environmental program and financial matters associated with the site.

1.2.2 Project Manager

Mr. Robert Raymond, URS Diamond, will serve as the project manager for this project. As Project Manager, Mr. Raymond is responsible for implementing the project and has the authority to commit the resources necessary to meet project objectives and requirements. He will work closely with the project technical resources and report directly to the Project Director. Mr. Raymond's responsibilities will include the following:

- ❏ Assigning duties to the project team and orienting the team to the needs and requirements of the project
- ❏ Disseminating project-related information from DuPont
- ❏ Serving as a liaison with subcontractor organizations, unless specifically delegated to others
- ❏ Interacting with the Quality Assurance Officer and Health and Safety Officer to ensure that these programs are functioning effectively
- ❏ Serving as the collection point for the project team's reporting of nonconformance with QA procedures or changes in project scope, documents, and activities

1.2.3 Project Hydrogeologist

The Project Hydrogeologist will be James Richendorfer, PhD, URS Diamond. His responsibilities will include the following:

- ❏ Technical lead for the groundwater investigation program
- ❏ Developing workscopes and evaluating site analytical data
- ❏ Overseeing and managing the preparation of all groundwater assessment reports

1.2.4 Technical Resources

A variety of technical resources will participate in the development and implementation of sampling programs associated with the site characterization activities. The technical resources will include, but are not limited to:

- ❏ Mr. Mark Vetter (DuPont CRG) – geology/hydrogeology
- ❏ Ms. Kimberly Johnson (URS Diamond)- geology/hydrology
- ❏ Ms. Nancy Grosso (DuPont CRG) – geology/hydrogeology and conceptual modeling
- ❏ Ms. Maryann Nicholson (DuPont CRG) – risk assessment and ecological assessment

Technical resources will provide strategic-level direction for all sampling programs and participate in the review of project-specific technical documents.

1.2.5 Quality Assurance Officer/Project Chemist

Ms. Sharon Nordstrom, URS Diamond, will serve as the project's Quality Assurance Officer/Project Chemist. Her responsibilities will include the following:

- ❑ Developing and administering the Quality Assurance Plan
- ❑ Reviewing analytical data generated for the project
- ❑ Assisting in day-to-day QA activities
- ❑ Interacting with subcontract analytical laboratories and validation firm regarding scheduling, technical, and QA issues

1.2.6 Health & Safety Officer

Ms. Kathy Sova, URS Diamond, will serve as the project Health and Safety Officer. Her responsibilities will include the following:

- ❑ Developing, reviewing, and implementing the project's Health and Safety Plan
- ❑ Ensuring that the project Health and Safety Plan is consistent with all applicable state and federal regulations

1.2.7 Field Services Manager

Mr. Jon Hammerberg, URS Diamond, will coordinate and lead all field sampling activities at the Barksdale site. He will also be responsible for interacting with the Project Chemist regarding sampling schedules and requirements, and oversee the packaging and shipping of samples to off-site laboratories.

1.2.8 Analytical Laboratory Subcontractors

It is anticipated that SevernTrent Laboratories (STL), Denver, Colorado will serve as the primary subcontract laboratory. STL-Denver has performed the analysis for previous well monitoring rounds at the site and is familiar with the project's technical and quality assurance requirements. STL-Denver has been approved by the Wisconsin Department of Natural Resources (DNR) for the analytes of concern at this site. Additional information regarding the laboratory qualifications, certifications, operations, and organization is provided in Appendix B. The STL-Denver Project Manager and point-of-contact will be Ms. Gail Deruzzo.

She will be responsible for the following:

- ❑ Ensuring that the project's technical and contractual requirements are relayed to laboratory management and operations personnel
- ❑ Tracking project deliverables
- ❑ Coordinating with the Project Chemist and field personnel on a daily basis

The STL laboratory facility in Sacramento, California may be used as a backup laboratory for the analysis of explosives on an as-necessary basis. In addition, EnChem, Inc.'s laboratory in Madison, Wisconsin will be used for the analysis of waste

characterization and short turn-around screening analyses. Because of their local proximity to the Barksdale facility, EnChem will also be used for the analysis of samples that exceed the 1% explosive hazard threshold for shipping samples from DuPont sites via air transportation (refer to Section 5). EnChem's Madison Laboratory has also been approved by the Wisconsin DNR for analysis of the target analytes.

1.3 Communication Pathways and Procedures

The Project Manager will be the focal point for all project communication and problem resolution. Issues related to field sampling and on-site activities will be relayed to the Project Manager via the Field Services Manager. Issues concerning the laboratory analysis of project samples or data quality will be transmitted to the Project Manager by the Quality Assurance Officer/Project Chemist. It will be the responsibility of the Project Manager to keep the Project Director informed of any problems or issues involving scope, budget, or significant technical issues. The Health and Safety Officer will be immediately advised of any issues, concerns, or incidents involving personnel safety and welfare.

1.4 Modifications to Approved QAPP

All modifications to the sampling procedures, analytical procedures, data assessment, and/or reporting will be submitted for approval using QAPP addendums. These addendums will include an approval/signoff page, similar to the original QAPP that will encompass key project personnel.

All key management, as outlined in Section 1.3, will have the authority to initiate and request QAPP modifications. All preliminary modifications will be orchestrated through the Quality Assurance Officer, who will compile and format the addendum and submit it to the project management team and the Wisconsin DNR for approval. Following agency approval, the Quality Assurance Officer will be responsible for distributing the addendum to the project team members.

2.0 PROJECT PLANNING AND DEFINITION

2.1 Project Planning Meetings

Internal meetings will be conducted prior to the implementation of all field activities. The internal meetings will include the appropriate team members referenced in Section 1.3, and will be conducted to review the project's specific tasks and responsibilities. Health and safety procedures and waste management procedures will also be reviewed.

2.2 Problem Definition/Site History/Background

The former DuPont Barksdale Works initiated dynamite production in 1904. Located on an 1,800 acre site in Bayfield County, Wisconsin, south of Washburn, the former plant was situated on Chequamegon Bay, Lake Superior.

During World War 1, the Barksdale facility was world's largest source of trinitrotoluene (TNT), producing 130 million pounds of the explosive between 1913 and 1918. Production was scaled down in the years following the end of the war, and then increased again during World War II, with an estimated 226 million pounds of TNT produced for the war effort. Once the war ended, production was again decreased to a level necessary to support regional mining needs.

DuPont ceased operations at the Barksdale Works in 1971, and most of the buildings existing at the time were subsequently demolished. The Barksdale property was sold in 1986 to Bretting Manufacturing Company, Inc. of Ashland, Wisconsin. The former main manufacturing area is currently operated as a private game reserve.

Limited soil and groundwater sampling and analysis have been conducted at the site since DuPont issued the *Site Conditions Report* by in 1997. Previous Investigations of the groundwater quality at the property and surrounding vicinity are summarized in the *Groundwater Investigation Report* (issued in December 1998), and the *1999 Groundwater Monitoring Report* (issued in March 2000). In addition, routine monitoring of residential wells in the vicinity of the property has been conducted since 1997.

2.3 2001 Sampling Programs

As discussed in Section 1.0, this QAPP has been developed to provide sampling, analytical, and quality assurance procedures for all groundwater, surface water, soil, and sediment sampling activities to be conducted at the site. The primary sampling events planned for 2001-2002 described in the following sections.

2.3.1 Burning Ground Investigation

The Burning Ground Investigation, initiated in Summer 2001, focuses on an investigation of the groundwater, soil, and sediment conditions in the former Nitroglygerin Burning Ground (NGBG), located along an intermittent stream in the central portion of the former Barksdale Works grounds. The sampling effort, as detailed in the *Draft Burning Ground*

Investigation Work Plan (March 2001), specifically addresses soil sampling in the former NGBG area; sediment sampling along the site drainage between the NGBG and monitor well PZ-1; and grab sampling of ground and surface water in the drainage areas and below the NGBG.

Samples will be collected in accordance with the protocols established in the project work plans, and as further described in Section 5. Soil, sediment, and surface water (pond and stormwater runoff) samples will be analyzed for target Nitroaromatic and Nitroamine Explosives, Appendix IX Volatiles, Appendix IX Semivolatiles (BNA), and Appendix IX metals. Temporary wells established in the Burning Ground area will be sampled for Target Explosives and Appendix IX Volatiles. Specific analytes and associated reporting limit requirements are summarized in Tables 8-1 through 8-8.

2.3.2 SUPPLEMENTAL GROUNDWATER INVESTIGATION

The Supplemental Groundwater Investigation, also initiated in Summer 2001, focuses on the further investigation of low level 2,4 and 2,6-DNT detections in the groundwater near the former Barksdale Works. The work effort includes installing four stream gauging stations to monitor flow along the intermittent creeks, and installing, developing, and sampling XX new monitor wells to evaluate vertical groundwater quality in the vicinity of monitor well PZ-1 (results of the 1999 groundwater investigation indicated order-of-magnitude higher concentrations of 2,4- and 2,6-DNT at this location), at the northern property line, and along the intermittent site drainages.

This investigation also includes an expanded monitoring of private residential wells in the vicinity of the Barksdale property. A number of these wells have been sampled on an on-going basis since 1997, when low concentrations of explosives were detected and carbon filtration systems were installed.

All wells (monitor and residential) will be sampled for the Nitroaromatic and Nitroamine Explosives. Additional water-quality indicator parameters, metals, and the Wisconsin-regulated volatile compounds will be analyzed for locations not previously sampled (newly installed monitor wells) and selected residential locations.

3.0 PROJECT DESCRIPTION AND SCHEDULE

As indicated in previous sections, this QAPP specifically addresses the groundwater/surface water and soil/sediment sampling anticipated to be conducted in 2001. The specifics of the sampling efforts are described in Section 2.3. Activities not specifically addressed in the QAPP will be communicated to the Wisconsin DNR as they are developed.

4.0 PROJECT QUALITY OBJECTIVES & MEASUREMENT PERFORMANCE CRITERIA

4.1 Project Quality Objectives

Sampling events associated with this QAPP will assist in evaluating potential impacts to groundwater, surface water, and sediments from past site activities. The analytical results from this sampling event, as well as results from previous investigations will be used to identify areas of potential groundwater, surface water, and soil contamination. Analytical results will also be used to direct future investigations at the site. The installation and sampling of additional monitor wells, and the expanded sampling of area residential wells will be used to evaluate vertical groundwater quality at the northern property line, along the intermittent site drainages, and in specific areas previously determined to have elevated levels of explosives contamination.

Additional project quality objectives include the further characterization of water quality adjacent to Highway 13 (Barksdale Village and Bretting residences), water quality adjacent to the Nolander Road residences, hydrogeology in the east-central portion of the plant, and the identification of potential sources of contamination.

4.2 Measurement Performance Criteria

To ensure that the data generated during the sampling programs is consistent with the stated project quality objectives, Measurement Performance Criteria for both field- and laboratory-generated data have been established and are presented in Tables 4-1 and 4-2. These criteria address the specific field and laboratory quality control checks that will be performed and/or quality control samples that will be analyzed to determine compliance with the following Data Quality Indicators (DQI): Precision, Accuracy/Bias, Completeness, Representativeness, Comparability, and Sensitivity. These indicators, as related to the project objectives are discussed below:

- **Precision** is defined as the agreement between numeric values for two or more assessments that have been obtained in an identical manner. Precision will be quantitatively assessed through the evaluation of % relative percent difference (RPD) values for Laboratory Control Sample (LCS) LCS duplicates (LCSD) pairs, matrix spike/spike duplicate pairs, field duplicates, and laboratory replicates.
- **Accuracy/Bias** is defined as the degree of agreement of a measurement with its accepted or true value. Accuracy will be quantitatively assessed for this project through the evaluation of the percent recovery of LCS samples, matrix spike samples, and sample surrogates.
- **Completeness** is a percentage of the quantity of valid data obtained from the measurement system compared to the quantity that was expected based on the sampling plan. Project completeness will be determined following the evaluation of data generated for each sampling program.

- **Representativeness** qualitatively expresses the degree to which the sample collection and analytical protocols adequately reflect the environmental conditions present at the specific sampling location, and the degree to which the sampling locations reflect the conditions of the site. The collection and analysis of field duplicate samples, review of current monitor well positions and sampling depths, and evaluation of newly obtained results as compared with historical data will be used to assess representativeness.
- **Comparability** expresses consistency in sampling and analytical procedures so that one data set can be compared to another. For this project, all measurement data will be calculated and reported in units consistent with standard practice to allow comparability of data. In addition, sampling procedures, water level readings and other field measurements, and where possible, field-sampling personnel will be consistent for all sampling events.
- **Sensitivity** is the ability of the method and/or instrument to detect the contaminants of concern and other target compounds at the level of interest. Quantitative measurement performance criteria need to ensure that the quantitation limits can be routinely achieved for each matrix, analytical parameter, and concentration level. All subcontract laboratories will be required to submit the results of current MDL studies for the target analytes prior to receiving and analyzing project samples.

5.0 SAMPLING PROCEDURES AND REQUIREMENTS

5.1 Sampling Procedures and Modifications

The Field Services Manager or a senior member of the field team will be responsible for overseeing all sampling activities and coordinating the transportation of samples to off-site laboratories. The subcontract laboratory will provide the necessary sample containers with preservatives, labels, custody forms, and shipping containers (coolers).

At a minimum, the following activities will be performed during the pre-sampling planning process:

- ❏ Review the sampling and analysis plans/resisions to determine and identify sample locations, matrices, and methods for sample collection.
- ❏ Coordinate with laboratory staff to ensure capacity and on-time delivery of sample containers and laboratory-provided supplies to the site.
- ❏ Arrange for secure storage of sample containers and supplies at the site prior to and during each sampling event.
- ❏ Determine that the appropriate log books, etc. are available for use during the sampling event.
- ❏ Assign accountability and responsibility for each activity to be performed by the field sampling team.
- ❏ Conduct a preliminary inspection, inventory, and cleaning of field equipment and instrumentation.
- ❏ Ensure that preventative maintenance on field equipment/instrumentation is up to date.
- ❏ Calibrate and re-check field instrumentation in accordance with Standard Operating Procedures (SOPs).
- ❏ Review approved QAPP and sampling plan requirements with field team members.
- ❏ Review the approved Health and Safety Plan for health and safety requirements and for determining the protective equipment that will be needed to conduct the sampling effort.

Field equipment will consist of the following:

- ❏ Laboratory-provided sample containers and supplies
- ❏ Field sampling logs/notebooks (example page in Appendix A)
- ❏ Wet ice for packing samples following sample collection
- ❏ Nonphosphate glassware detergent

- ❑ Laboratory-supplied deionized/distilled water for rinsing field equipment and equipment blanks
- ❑ Orion Model 250A pH/Temperature Meter; Orion Model 115 Conductivity Meter; and Orion 835 Dissolved Oxygen Meter for on-site field measurements
- ❑ Photoionization detector (PID) for organic vapor analysis
- ❑ Waders or waterproof boots for surface water sample collection
- ❑ Low-flow dedicated bladder pumps with a ⅜-inch, Teflon-lined polyethylene tubing for monitor well purging and sampling
- ❑ Oil-less Gas Compressor
- ❑ Electronic water level probe
- ❑ 0.45 micron disposable filtering cartridges for field-filtering metals in groundwater samples
- ❑ Disposable Teflon bailers for surface water/temporary well grab samples
- ❑ For collection of soil and/or sediment samples: 5 gallon buckets, stainless steel hand augers or Ponar-type grab sampler, stainless steel scoops and/or trowels, stainless steel bowls, and aluminum foil.

5.2 Groundwater Sampling Protocol

Preparing for groundwater sampling events will include acquiring all of the necessary monitoring equipment listed above and a review of pertinent site information . Prior to each sampling event, a complete round of depth to water levels will be measured to the nearest one hundredth of a foot. Each of the wells will have a permanent mark placed on top of the well casing (if not already present) to identify the surveyor's reference point and to standardize the measuring point for depth to water measurements. Water levels will be measured with an electronic water level probe. The probe will be decontaminated between wells as specified in Section 5.8.

The sampling pump will be placed into each well with the intake at approximately the level of mid-screen or slightly above, with as little turbulence as possible. Prior to collection of the samples for analysis, all wells will be purged using a low-flow (minimum drawdown) protocol with purge rates ranging from less than 0.1 L/minute to 0.5 L/minute. Low-flow bladder pumps with dedicated tubing will be used to evacuate the groundwater from the screen area of the well. Purgewater will be managed in accordance with the site's Waste Management Plan. The water quality parameters (pH, specific conductivity, dissolved oxygen, and temperature) will be monitored using a flow-through cell. Purging will be considered to be complete and formation water accessed when all field measurements have stabilized. Stabilization is considered to be achieved when three consecutive sets of field readings, taken at 3-5 minute intervals are within 10% of each other

Following well stabilization, the purge rate will be lowered, and the groundwater samples will be collected directly from the dedicated discharge tubing. Individual sample aliquots will be collected in the following order: volatiles, semivolatiles, explosives, metals, and

inorganics. Groundwater sample aliquots for dissolved metals will be filtered at the time of sample collection through an in-line 0.45 micron membrane filter cartridge prior to preservation. Grab samples from temporary, or newly installed wells or piezometers may be collected for screening analyses using disposable Teflon bailers.

Sample locations designated for the collection of matrix spike/spike duplicate samples will require two additional sets of sample containers to be filled. Sample locations designated for the collection of field duplicate samples will require one additional sample container set to be filled. Immediately following sampling, all sample containers will be checked for labeling accuracy and placed in iced coolers for storage and shipment.

5.3 Residential Well Sampling Protocol

Residential well samples will be sampled directly into the laboratory-provided sample containers from house taps or faucets without aerators, hoses, or water filtration/purification equipment. Residential wells with DuPont-supplied carbon filter systems will be sampled at the inflow, mid-system, and the outflow. Residential well locations designated for the collection of field duplicate samples will require one additional set of sample containers to be filled. Sample locations designated for the collection of matrix spike/spike duplicates will require two additional sets of bottles to be filled. Immediately following sample collection, the labeling on all sample containers will be checked for accuracy, and the samples will be placed in coolers with wet ice for transport.

5.4 Surface Water Sampling Protocol

Surface water samples may be collected directly into the sample containers when the surface water source is accessible on foot or by wading, taking care to disturb the bottom sediment as little as possible. For pre-preserved sample containers (volatile organic analysis vials and metals) or pond sampling locations that are not easily sampled directly into the sample containers, a sample transfer container or Teflon bailer will be used to prevent loss of the preservative and to ensure zero head space in the final sample containers. Sample transfer bottles will be disposable (single-use) or will be decontaminated as described in Section 5.8 prior to sample collection. Immediately after sample collection, samples will be labeled and transferred to a cooler with wet ice for temporary storage. Prior to or immediately subsequent to sample collection, field measurements for pH, specific conductivity, and temperature will be taken and recorded in the field log books. Samples designated for the collection of field duplicates will require one additional set of sample containers, and matrix spike/spike duplicate samples will require the collection of two additional sets of sample containers.

5.5 SOIL SAMPLING PROTOCOL

Soil samples will be collected from trenches, surface locations, and boreholes using stainless steel hand augers, stainless steel scoops, or trowels. Samples will be transferred into stainless steel bowls, mixed and quartered, and aliquots transferred into sample containers. Samples for volatiles analysis will be collected directly into 5 gram

disposable (single use) EnCore samplers (three samplers per location). Sample locations designated for the collection of matrix spike/spike duplicate or field duplicate samples will require at least one additional set of bottles and three additional EnCore samples to be filled for each. All soil sampling equipment will be decontaminated prior to and between the collection of each sample in accordance with Section 5.8. Sample residuals will be managed in accordance with the site's Waste Management Plan. Samples will be stored in coolers with wet ice until prepared for shipment.

5.6 Sediment Sampling Protocol

The physical location of the sediment sampling point and nature of the substrate may dictate the type of sampling equipment that will be used. Sediment sample collection will be performed by excavating a sample using a stainless steel hand auger or similar coring device, with a stainless steel scoop, or with a stainless steel Ponar-type grab sampler. At locations with shallow water, a PVC bucket with the bottom removed may be used to temporarily isolate the sediment sample extraction area from the surface water to minimize the loss of fines. Prior to conducting field activities, all sediment sampling equipment will be decontaminated as described in Section 5.8. Samples will include the full sediment depth, and end within the underlying native soil.

Sample aliquots for volatiles analysis will be collected in a manner that minimizes disturbance of the sample and transferred directly into pre-labeled 125gram wide-mouth, glass sample containers (5 gram EnCore samplers will be used for dry- or well-consolidated sediments). For non-VOC samples, multiple cores may be needed to provide sufficient sample volume to meet the analytical requirements. If so, multiple cores will be collected from the same location and homogenized by quartering and mixing in stainless steel bowl with a stainless steel scoop or trowel prior to transfer to sample jars. Sample residuals will be managed in accordance with the site's Waste Management Plan. Samples will be stored in coolers with wet ice until prepared for shipment.

5.7 On-site Soil Screening

On-site screening of soils for explosives with Immunoassay test kits has been used at Barksdale during previous site investigation work to delineate areas of potential contamination and identify areas for further characterization. Strategic Diagnostics, Inc. (SDI) Dtech Immunoassay kits will be used to provide semi-quantitative screening of soils/sediments to be collected in areas with known or suspected high concentrations of target explosives. In accordance with DuPont policy, samples screening higher than a 1% concentration (100,000 ppm) will not be shipped for off-site laboratory analysis via air transportation. Samples with screening concentrations between 1-3% will be transported for off-site analysis only via ground transportation (direct laboratory courier), and any sample screening higher than 3% will not be transported off-site.

5.8 Decontamination, Calibration, and Inspection of Sampling Equipment

Disposable or dedicated sampling equipment will be used wherever possible. When this is not applicable, the following field decontamination protocol will be implemented for field equipment:

- ❏ Wash with nonphosphate glassware detergent plus tap water.
- ❏ Thoroughly rinse with tap water.
- ❏ Thoroughly rinse with distilled/deionized water.

Non-dedicated pumps will be cleaned and flushed prior to and between each use using the following protocol:

- ❏ External detergent wash
- ❏ Tap water rinse
- ❏ 10-20 gallon (depending on diameter of pump) flush of potable water through pump
- ❏ External distilled/deionized water rinse

5.9 Inspection and Acceptance Requirements for Field Supplies and Sample Containers

New, pre-preserved sample containers (I-Chem 200 Series or equivalent), and 5 gram disposable EnCore samplers will be used for the collection of all field samples, matrix spike/spike duplicate samples, equipment blanks, and trip blanks. Individual sample container requirements are itemized in Table 6-1. Distilled/deionized (DI) water for final decontamination rinsing and collection of equipment blanks will be transported to the site in 2-liter glass containers. All sample containers, deionized water, and trip blanks will be provided by the subcontract laboratory performing the analysis. Documentation of sample container and DI water lots will be maintained by the laboratory. All sample containers and supplies shipped directly to the site will be delivered at least one business day prior to the scheduled day of sampling to allow for time to inspect the shipment for damage, shortages, incorrect items, and request replacements. All sample containers and supplies will be shipped to the site under chain-of-custody documentation.

5.10 Waste Disposal Procedures

Site-specific Waste Management Plans have been prepared to address on-site segregation, storage, and disposal of anticipated waste materials. These will include (but are not limited to) personal protective equipment (PPE), decontamination water, and groundwater and soil/sediment wastes from investigation activities. All wastes generated during monitor well drilling and sampling will be containerized in appropriate DOT-approved storage containers, labeled, and retained on-site pending completion of any required waste characterization analyses. Any changes in or additions to waste handling procedures will be detailed in a Waste Management Plan Addendum.

6.0 SAMPLE HANDLING, TRACKING, AND CUSTODY REQUIREMENTS

6.1 Sampling Methods Requirements

Minimum sample volumes, container types, preservative requirements, and holding times for the required laboratory analyses are listed in Table 6-1.

6.2 Documentation of Sample Collection/Sample Handling Procedures

The procedures outlined in this section ensure that individual samples are tracked and handled from the time of field collection through laboratory analysis. Each sample container will have a sample label affixed to the outside specifying the sample identification number, location sampled, preservatives (if any), and parameters to be analyzed. Sample labels are typically initiated in the laboratory and affixed to the individual containers prior to shipping to the field, and any additional information added at the point of sample collection. All information will be recorded on the sample label with waterproof ink.

Following collection and labeling, samples will be stored on wet ice until packed for shipment to the laboratory. The Field Services Manager will coordinate arrangements for sample transportation with the Project Chemist. All samples will be transported to the laboratory (either via laboratory courier pick-up at the site or commercial carrier) in sealed coolers or sample shuttles filled with wet ice. Volatile organic analysis (VOA) vials will be packed in foam sleeves to protect against breakage. Samples will be packaged in Ziplock plastic bags. Special precautions will be taken if the sampling must be conducted during extreme cold weather to prevent the samples from freezing. Additional instructions for packing samples for shipment are provided in SOP S-2 in Appendix A.

The field sampler will log the appropriate sample collection information for each sample location, noting the following information:

- ❏ Sample location
- ❏ Sample identification number (based on Sample Identifier Protocol included in SOP S-3-DuPont CRG Standard Operating Procedure for Completing Chain-of-Custody Forms)
- ❏ Sample source (e.g., monitoring well) and method of sample collection
- ❏ Evacuation date and time
- ❏ Volume purged (note if purged to dry)
- ❏ Sampling personnel
- ❏ Depth to bottom, depth to water, and casing volume

- ❑ Date and time of sample withdrawal if different from time of purging
- ❑ Sample appearance (e.g., color, turbidity, odor, presence of sediment)
- ❑ Weather conditions at time of sample withdrawal
- ❑ Field measurements for aqueous samples
- ❑ Number, size, type of containers, preservatives, and required analyses for each sample aliquot
- ❑ Comments and observations at time of sample withdrawal
- ❑ Signature and date of field log book completion

6.3 Field Custody Procedures

As indicated in Section 6.1, the chain-of-custody (COC) record will normally be initiated in the laboratory at the time the sample containers are prepared for shipment to the field and will accompany the empty containers to the site. The following information will be recorded on the COC record prior to shipping samples from the site to the laboratory:

- ❑ Collector's name
- ❑ Dates and times of sample collection
- ❑ Sample identification numbers
- ❑ Number of containers for each sample aliquot (if not already indicated)
- ❑ Container size/type (if not already indicated)
- ❑ Type of preservation (including ice)
- ❑ Parameters (analytes from each sample aliquot)
- ❑ Turn-around requirements
- ❑ Special handling instructions
- ❑ Destination of samples
- ❑ Name, date, time, and signature of each individual possessing the samples

The COC record will be signed by each individual responsible for custody of the sample containers. Custody of the samples will be defined as actual physical possession, in view after physical possession, or sealed in a tamper-resistant container after physical possession. The original signed COC record will accompany the samples to the laboratory and be returned as part of the final data package. The Field Services Manager or designate will be responsible for custody of the samples until transfer to the laboratory. All coolers will be sealed with tamper-evident tape or a tamper-evident seal prior to leaving the field site. Additional instructions for completing chain of custody records are provided in SOP S-3 in Appendix A.

6.4 Laboratory Custody Procedures

The laboratory sample custodian is responsible for inspecting and verifying the correctness of COC records when received and for verifying that all samples are received. The custodian is also responsible for verifying sample temperatures at the time of receipt and that the sample containers are appropriately preserved (VOA vials will not be opened to check preservation). Upon sample receipt, the sample custodian will sign the COC record and note the sample conditions on it. The sample custodian will also immediately notify the laboratory project manager of any discrepancies, damaged or broken sample containers, or evidence of tampering (e.g., broken custody seal), who will in turn immediately notify the Project Chemist. Confirmation of sample receipt at the laboratory will be submitted to the Project Chemist for all incoming sample delivery groups.

As samples are received at the laboratory, they will be entered into a sample management system. The following minimum information will be provided:

- ❏ Laboratory sample number/identification
- ❏ Field sample designation
- ❏ List of analyses requested for each sample container

Immediately after receipt, samples will be transferred to a secure storage area with appropriate temperature control (as established by EPA document SW-846, Third edition, Chapters 2, 3, and 4) to await preparation and analysis. Only authorized laboratory personnel will have access to the locked storage area(s). The laboratory will have in place internal sample security procedures and is also responsible for the proper management and disposal of all sample residuals following all applicable federal, state, and local laws, rules, and regulations (unless alternate arrangements are made for the return of sample residuals to the Barksdale site for disposal).

7.0 FIELD ANALYTICAL METHOD REQUIREMENTS

7.1 Field Analytical Methods and Modifications

Table 4-1 summarizes the field measurements, analytical methodologies, and associated reporting limits for the project.

7.2 Field Analytical Instrument Calibration and Inspection Requirements

Instrument calibrations (both field and laboratory) typically consist of two types: initial calibration and continuing calibration. Initial calibration procedures establish the calibration range of the instrument and determine instrument response over that range. Several different analyte concentrations (standards) are used to establish instrument response over a concentration range. The instrument response over that range is commonly expressed as a correlation coefficient or by a response factor. Continuing calibration, when required, usually includes the measurement of a single standard. The response is compared to the initial measured instrument response. Calibration procedures, frequency, and acceptance criteria for the field instruments to be used for this project will be based on manufacturer operating instructions and DuPont/URS Diamond field policies.

The Field Services Manager is responsible for supervising field instrument calibration and maintenance and ensuring adherence to the calibration schedule and each operator's understanding of the proper usage, maintenance, and storage of each instrument. A calibration log book is kept with each instrument to record the field calibration. Each log book contains the date of calibration, the operator's initials, the calibration measurements, and observations about the instrument or calibration procedures. Any piece of equipment that is not operational or appears to be malfunctioning will be removed from service, tagged, and segregated for repair or replacement.

7.3 Field Corrective Action

Corrective action in the field may be necessary when field conditions change or when sampling procedures and/or field analytical procedures require modification. In general, the field team member, Field Services Manager, Project Hydrogeologist, Project Chemist, or Project Manager may identify the need for corrective action, as well as recommend appropriate corrective action measures. Correction action for field measurements may include the following:

- ❏ Repeating the measurement
- ❏ Checking all proper adjustments for ambient conditions
- ❏ Checking instrument batteries
- ❏ Checking instrument calibration and/or recalibration

- ❑ Replacing instrument or measurement device
- ❑ Stopping work (if necessary)

8.0 FIXED LABORATORY ANALYTICAL METHOD REQUIREMENTS

8.1 Fixed Laboratory Analytical Method Requirements, SOPs and Modifications

Method selection criteria for the project are based on analytical methods used in previous investigations of the site and by the Project Data Quality Objectives, coupled with the method detection limits achievable for each of the target analytes (contaminants of concern). Analytical methods have been selected to address the intended use of the data in a timely and cost-effective manner.

Tables 8-1 through 8-5 list the target analytes, analytical method references, and associated reporting limits required for this project. The STL-Denver SOP for Method 8321A, and supporting documentation are included in Appendix C. Any modification or changes in analytical methodologies, as well as the reason for the change, will be communicated to the Project Chemist and documented in all associated correspondence and records. The modifications will also be identified in the laboratory data package. Any missed holding times must be reported to the Project Chemist as soon as identified and also documented in the final data package.

The project reporting limits are based on current laboratory method detection limit (MDL) data. All results will be reported to the MDL, with a data qualifier added to any analyte with a concentration between the MDL and project reporting limit. Individual sample reporting limits may vary due to dilution requirements, variability in the sample volume used to perform the analysis, the presence of analytical background contaminants, matrix interferences, or other sample- or analysis-related conditions. All soils/sediments will be reported on a dry-weight basis.

Particular attention to reporting limits are required for the analysis of low-solids sediments. During the sampling of sediments, every effort will be made to minimize excess liquid in the field. If possible, laboratory personnel should decant the standing water from sediment containers prior to homogenization and weighing (care must be exercised to avoid loss of sediment fines). Increasing the amount of sample volume extracted or digested to compensate for percent moisture is recommended whenever possible. For samples with very low percent solids (<30%), the Project Chemist should be consulted to discuss alternative sample preparation procedures.

8.2 Fixed Laboratory Instrument Calibration, Maintenance, and Inspection Requirements

As with field analytical instruments, calibration of fixed laboratory instruments typically requires the analysis of a series of initial calibration standards, as well as the periodic analysis of a single (or smaller series) continuing calibration standard.

Calibration standards are chosen to bracket the expected concentrations of target analytes in the sample and to operate within the linear response range of the instrument. Sample concentrations that fall above the calibration range are diluted and reanalyzed until they are within the calibration range. Organic analyses are typically quantitated from five-point initial calibration curves. During the course of analysis, additional calibration standards are routinely analyzed to ensure that the instrumental response has not exceeded the method acceptance limits. The continuing calibration criteria specified by the method or laboratory SOPs are used by the analyst to determine whether the instrument must be re-calibrated or whether the instrument conditions can be further optimized. The accuracy of working standards is verified by comparison with a standard from an independent source. All organic standards must be refrigerated or frozen, as specified in the applicable analytical method.

Fixed laboratory instrument calibration procedures acceptance criteria will be performed as described in the referenced analytical method and as detailed in the individual laboratory SOPs and Quality Assurance Plan.

8.3 Fixed Laboratory Corrective Action

Both STL-Denver and EnChem have quality systems in place that include a deficiency reporting mechanism. The system includes documentation of the deficiency, implementation of both immediate and long-term corrective actions, and immediate notification of the Project Chemist of any deficiencies, errors, out-of-control events, or unusual occurrences that impact the quality of the sample data.

An out-of-control event is defined as any event that is beyond the acceptance limits established for laboratory operation by the Laboratory SOPs, EPA method, or contract requirements. An out-of-control event can be due to data that are outside the acceptance limits for accuracy and/or precision, method blank contamination, improper instrument calibration or maintenance, or deviations from the SOP detected by a QA audit.

An unusual occurrence is a situation in which the analytical system is compliant with the protocol or SOP and is, therefore, in control, but an atypical or undesirable incident has occurred that warrants further investigation. Such an occurrence could be a contaminated holding blank or difference in the pattern of non-spiked target compounds between spiked and unspiked aliquots of a sample used as the matrix spike.

Any errors, out-of-control events, or unusual occurrences will be documented in the laboratory batch files and in the final data report.

8.4 Fixed Laboratory Inspection and Acceptance Requirements for Supplies

Procedures for the selection of vendors, purchase, storage, use, and disposal of standards, reagents, and expendable materials required for laboratory operations are addressed in the laboratory Quality Assurance Plans and SOPs.

Method selection criteria were determined by analytical methods used during previous investigations and by the project quality objectives, coupled with the method detection limits achievable for the contaminants of concern.

9.0 QUALITY CONTROL REQUIREMENTS

Quality Control is an integrated system of activities in the area of quality planning, quality assessment, and quality improvement designed to provide the project with a measurable assurance that the required standards of quality are being met. The QC checks/samples to be implemented for this project fall into two general categories: those QC checks analyzed on an individual basis and those that are performed with a specific set or "batch" of samples.

Batch-specific QC checks include the QC samples that are handled, prepared, and analyzed with the investigatory samples to ensure that the sampling, transportation, and analytical procedures are performed under known, well-defined conditions. The batch-specific QC samples to be collected and/or analyzed for this project include trip blanks, equipment blanks, laboratory method blanks, matrix spikes, laboratory control samples, and calibration check standards.

Sample-specific QC checks include the QC samples used to evaluate potential sources of error in the collection, transportation, and analysis of individual samples. Sample-specific QC checks to be analyzed for the project include sample surrogate spikes and internal standards.

9.1 Sampling Quality Control

The types and frequency of field QC samples to be collected for the project are summarized in Table 4-1.

9.2 Analytical Quality Control

Analytical Quality Control Checks to be implemented for the project are summarized in Tables 4-1 and 4-2.

9.2.1 Analytical Quality Control Checks

The types and frequencies of quality control checks analyzed with each sample are defined in USEPA SW-846 3rd Edition, Update III, 1996, and are discussed below:

- **Surrogate** (organics): Each sample, matrix spike, matrix spike duplicate, and blank are spiked with surrogate compounds prior to purging and extraction in order to monitor preparation and analysis. Surrogates are used to evaluate analytical efficiency by measuring percent recovery.
- **Matrix Spike**: A matrix (soil or water) is spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring percent recovery of the spiked analytes.
- **Duplicate** (matrix spike duplicate or LCS duplicate): A second aliquot of a spiked matrix/sample is prepared and analyzed at the same time as the original sample

for the purpose of determining the precision of the method. Recovery of the original compared to the duplicate is expressed as relative percent difference (RPD).

- **Replicate**: A second aliquot of a sample is prepared and analyzed at the same time as the original sample for the purpose of determining the precision of the method. Recovery of the original compared to the replicate is expressed as relative percent difference.
- **Blanks** (method, preparation): Blanks are an analytical control consisting of a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples. (Metals use a digested reagent blank with soils.) They are treated with same reagents, internal standards, and surrogate standards and carried through the entire analytical procedure. The blank is used to define the level of laboratory background contamination.
- **Internal Standards** (GC/MS organics): Internal standards are compounds added to every standard, blank matrix, spike, matrix spike duplicate, and sample at a known concentration, prior to analysis. Comparison of the peak areas of the internal standards are used for internal standard quantitation as well as to determine when changes in the instrument response will adversely affect quantification of target compounds.
- **Serial Dilutions** (Inorganics GFAA and ICP): If the analyte concentration is sufficiently high, an analysis of a five-fold dilution must agree within 10% of the original determination. If the dilution analysis is not within 10%, a chemical or physical interference effect should be suspected.
- **Interference Check Sample** (ICP): To verify interelement and background correction factors, a solution containing both interfering and analyte elements of known concentration is analyzed at the beginning of each analysis run or a minimum of twice per 8 hours.
- **Laboratory Control Samples**: Aqueous and solid control samples of known composition are analyzed using the same sample preparation, reagents, and analytical methods employed for the sample. An LCS is typically analyzed with each sample batch and reported as percent recovery.
- **Calibration Check Standards**: The frequency of calibration and calibration verification, the number of concentrations used, and acceptance criteria vary for each analysis. Specific instrument calibration procedures are summarized in the laboratory SOPs and Quality Assurance Plans.

10.0 DATA ACQUISITION REQUIREMENTS (NON-DIRECT) MEASUREMENTS

As discussed in Section 4.0, a primary Project Quality Objective is the comparison of the analytical data to be obtained for the proposed sampling events with the historical data previously acquired for the site. The laboratory and field data obtained for the previous sampling events, as well as supporting documentation, field log books, project correspondence, and reports, are accessible in the project files. In addition, data obtained for sampling efforts since approximately 1995 have been evaluated and are stored electronically in the DuPont Corporate Environmental Database (CED).

11.0 DOCUMENTATION, RECORDS, AND DATA MANAGEMENT

11.1 Project Documentation and Records

Field and laboratory data collected during sampling events will be retained in both hardcopy and electronic format. Field analytical measurements will be transcribed directly from the field log books into the DuPont CED. All manual data entry will be checked for transcription errors prior to downloading. Field documentation, notes, and measurements not stored in the CED will be retained in the project files as discussed below. Specific laboratory deliverable requirements are addressed in Section 11.3.

The Project Manager or designee will be responsible for indexing and maintaining files for all field log books, field note books, project correspondence, any system or performance audits conducted, nonconformance reports, CED reports, etc. DuPont will maintain this archived information for a minimum of seven years following completion of the project. The subcontract laboratories will maintain all hardcopy documentation and records associated with the project in accordance with DuPont contractual requirements.

11.2 Data Management

11.2.1 Data Reduction

Data reduction involves the process of generating qualitative and quantitative sample information through observations, field procedures, analytical measurements, and calculations.

Data reduction occurs with:

- ❏ Pre-sampling planning, including the identification of sample locations and naming conventions
- ❏ The field sampling process through use of field logs and field measurements
- ❏ Field communications with the laboratory in sample analysis requests
- ❏ Field operations with collection, preservation, and COC documentation
- ❏ Laboratory operations with sample receipt, sample preparation and analysis, collation of raw data, and generation of final laboratory results
- ❏ Post-laboratory operations with tabulation of analytical results in a format suitable for reports, maps, and trend plots

11.2.2 Field Data Reduction

For field data measurements that require calculations to obtain final concentrations/values, the equations used and the calculations performed will be recorded

in the field note book. The field team member performing the field measurement will check all calculations at least once.

Occasionally, a field measurement will result in an outlier with a value significantly outside the expected range. During the field measurements, the field team, based on their experience, will attempt to identify outliers. When outliers are identified during a field effort, the outlier will be recorded as any other field measurement, the instrument will be checked and/or re-calibrated, and at least two additional measurements will be made and recorded to verify or invalidate the suspected outlier. If, after this check, the value remains the same, it is considered a valid measurement. If the value is determined to be invalid, the other measurements will be used.

11.2.3 Laboratory Data Reduction

In the laboratory, data reduction is performed by the analyst and consists of calculating the final analyte concentrations from the raw data or measurements. The complexity of the data reduction depends on the specific analytical method and the number of operations involved (e.g., dilutions and instrument readings). The analyst uses the raw data measurements to calculate the final reported result (usually with the assistance of computer programs). Copies of all raw data and documentation of the calculations used to obtain final results will be retained on file in the laboratory.

11.2.4 Data Review

Data review is the process of verifying that qualitative and quantitative information generated relative to a given sample is complete and accurate. Data review is conducted by an independent reviewer who is not involved in the actual processes of data reduction and reporting.

The DuPont quality control review process will be performed on 100% of the data received from the laboratory. This multi-step process begins with verification of laboratory reports against the associated chain-of-custody records to ensure completeness of all tests on all samples submitted and the verification of accuracy of electronic deliverables with hardcopy data reports. The process also includes verification of the representativeness of field sample results via review of associated field and trip blank data; determination of data usability by review of matrix QC and LCS results against project acceptance criteria; verification of quality control batch integrity to ensure method compliance; and verification of the Case Narrative documentation accompanying the reports.

As described further in Section 13, third- party, independent validation of the laboratory data deliverables is proposed for the 8321A Explosives data generated for the residential and key monitor well locations.

11.3 Data Deliverable Formats

Final data deliverables will be provided to URS Diamond as full documentation packages (CLP-type), except for waste characterization and rapid turn-around analyses, which will be provided in standard data report format. All final deliverables will be provided in

hardcopy and on diskette on a 21-day turn-around, unless otherwise requested . The data reported in the hardcopy deliverable and the electronic deliverable will be verified by the laboratory to be identical and will be formatted in accordance with DuPont's contractual requirements.

11.4 Sample Tracking

Sample tracking in the DuPont CED is initiated as soon as the project-sampling schedule is confirmed. Sample containers are requested from the laboratory via the Project Initiation Sheet, which is prepared by the Project Chemist and used as a means of notifying the laboratory of the final sampling schedule. The Project Chemist will be in daily contact with the field team during the sampling events and also with the laboratory. As samples are received in the laboratory, the Project Chemist will be notified of any problems, breakage, or discrepancies. Based on sample receiving dates, laboratory due dates are entered into the CED tracking system and monitored by the DuPont CRG/URS Diamond Analytical Data Quality Management (ADQM) group. When data deliverables are received from the laboratory, the electronic data is loaded, and the DuPont data review process is initiated. The review process is normally completed within seven days from receipt of the final laboratory data for the sampling event.

12.0 ASSESSMENTS AND RESPONSE ACTIONS

12.1 Additional QAPP Non-Conformances/Corrective Action

If, through the data evaluation process, problems or non-conformances with the QAPP are identified, corrective actions will be initiated. All identified QA problems and corrective actions will be documented to provide a complete record of QA activities and help identify needed long-term corrective actions.

The detection of system and performance problems and corrective actions used in the field during monitoring and sample collection will be documented in the field note books and on a Corrective Action Report to be submitted to the Project Manager. Any problems that cannot be resolved by the sampler or Field Services Manager will be brought to the attention of the Project Manager, Technical Resources, and/or Quality Assurance Officer/Project Chemist, for assistance and/or follow-up as appropriate.

The laboratory Quality Assurance Plans describe the corrective action procedures and documentation to be used by the laboratories. Any problems which cannot be resolved by the analysts, laboratory managers, and laboratory quality assurance personnel will be brought to the attention of the project Quality Assurance Officer/Project Chemist.

The laboratory personnel will assess laboratory QC samples and re-analyze stored samples that do not meet QC criteria prior to the expiration of the sample holding times. Corrective actions may include re-analysis, data qualification, or re-sampling and re-analysis. Laboratory Corrective Action Reports (CAR) will be used for documenting the identification and resolution of significant defects. The CAR forms will be kept on file in the laboratory QA files. A discussion of analytical exceptions and corrective actions implemented, if any, will also be included in report narratives.

12.2 QA Management Reports

Following the occurrence or identification of a specific problem, Quality Assurance reports summarizing specific Corrective Actions implemented will be submitted the Project Manager as soon as possible. Procedures for modifying the approved QAPP to reflect corrective actions or modifications in sampling or analytical protocols are discussed in Section 1.5.

Data reports issued for individual sampling events will include a summary of QA issues associated with the sampling and/or analysis of the sample set, as well as any corrective actions implemented.

13.0 DATA VERIFICATION REQUIREMENTS

Analytical data assessment will be performed by examining the results of data verification and data evaluation to determine the usability of the data for the project objectives. Data verification and data evaluation are separate levels of review that can be performed by themselves or in conjunction with each other. These levels of review are further defined in the subsections below. While the verification and evaluation processes can be applied to field data measurements, they are primarily associated with the review of laboratory data.

13.1 Data Verification

As discussed in Section 11, all laboratory data deliverables will be submitted in both hardcopy and electronic data formats. Upon receipt of the deliverables package, an ADQM staff member will load the electronic data deliverable (EDD) into the CED, and verify the following:

- ❏ Results were received for each requested analysis for each sample. If a result is missing, the staff member will determine whether the laboratory submitted a deficiency report to address the missing data.
- ❏ The data deliverable will be inspected for completeness based on the requirements specified in this plan. The inspection will verify that the report sections are present (but not necessarily the completeness of all data within the sections).
- ❏ The data reported in the electronic deliverable will be compared to the data reported in the hardcopy deliverable. The laboratory will be contacted to resolve and correct any discrepancies between the deliverables prior to further review.

13.2 Data Evaluation

Data evaluation will be performed by the Project Chemist to assess whether the quality control requirements for field duplicates, equipment blanks, trip blanks, surrogates, matrix spikes, laboratory method blanks, and laboratory control samples were met. Additional criteria including missed sample holding times, sample conditions (temperature, preservatives), and the case narratives will also be examined. This process will be performed on 100% of the project laboratory deliverables:

- ❏ If quality control outliers are observed in the evaluated data, the data point will be flagged, and the details of the exceedance will be added to the database as a comment.
- ❏ Method, equipment, and trip blanks are not expected to contain any target analytes with concentrations equal to or above the laboratory practical quantitation limit (PQL).
- ❏ Field duplicate results will be assessed based on the relative percent difference (RPD) between values, using the following equation:

$$RPD = \frac{(D1 - D2)}{(D1 + D2)/2} x100\%$$

where,

D1 = Primary sample result

D2 = Duplicate sample result

- Laboratory control spiked samples will be assessed based on the percent recovery (%R) of spiked analytes. The percent recovery is calculated using the following equation:

$$\%R = \frac{X}{TV} x100\%$$

where,

X = Observed value of measurement

TV = "true" value of spiked analyte

- Matrix spike/matrix spike duplicate (MS/MSD) and laboratory control sample (LCS) data will be assessed based on the percent recovery of spiked analytes using the following equation:

$$\%R = \frac{(SSR - SR)}{SA} x100\%$$

where,

SSR = Spiked sample result for analyte x

SR = Sample result for analyte x

SA = Spike added of analyte x

- The relative percent difference between MS/MSD or LCS/LCSD pairs will be calculated using the RPD equation presented above.
- Data completeness will be assessed based on the amount of valid data obtained from a particular measurement system (sampling and analysis). It may be quantitatively expressed using the following equation:

$$Completeness = \frac{N1}{N2} x100\%$$

where,

N1 = Number of valid measurements obtained

N2 = Number of valid measurements expected

In addition to the data evaluation process described above, all 8321 explosives data generated for residential well samples and key monitor well locations will be submitted for independent data validation.

Environmental Standards, Inc. in Valley Forge, Pennsylvania is proposed as the validation contractor for the project. A Data Validation Report will be produced by the validator for each data set examined, and data qualifier flags applied to individual results

as appropriate. The level of data review performed on each data delivery group (ADQM review or Full Validation) will be identified in the project database. Data validation will be performed in accordance with the guidance form the *National Functional Guidelines for Organic Data Review*, U.S. EPA, 2/94 and 10/99 update.

14.0 DATA USABILITY/RECONCILIATION WITH PROJECT QUALITY OBJECTIVES

Throughout the site investigation and groundwater monitoring programs, the Barksdale site project management team will determine if project quality objectives are being met and assess whether the data being collected are sufficient and appropriate.

Individuals making field measurements will determine whether field quality control criteria were met and assess the need for corrective action to be implemented in further sampling. This corrective action may include re-calibration of field instruments or use of a different type of instrument.

The analysts and supervisors in the laboratory will determine if analytical QC criteria are achieved and, if not, whether corrective action is warranted. As discussed in previous sections, the laboratory and field data will also be reviewed by the Project Chemist to determine usability with regard to the specific criteria established in the QAPP. Data may not always meet precision and accuracy requirements but may still be considered usable. The Project Technical Resources, in conjunction with the Project Manager and Project Director, will assess all data collected during the site investigation and groundwater monitoring programs and will advise Wisconsin DNR of any changes to the program that may be recommended as a result of data obtained during the sampling efforts completed to date.

15.0 REFERENCES

EPA, Office of Solid Waste and Emergency Response. June 1997. *Test Methods for Evaluating Solid Waste*. Laboratory Manual Physical/Chemical Methods, SW-846, Volumes 1A, 1B, and 1C. Third Edition. Washington DC.

EPA, October 1984. *Methods for the Chemical Analysis of Water and Wastes*. EPA-600/4-79-020

EPA, December 1992. *Specifications and Guidance for Contaminant Free Sample Containers.* EPA540/R-93/051

EPA, Office of Emergency and Remedial Response. October 1999. *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* EPA540/R-99/008.

TABLES

Table 4-1 Field Analytical Method Requirements, QC, and Calibration

Parameter Analysis Method	Achievable Sensitivity/ Lower Quantiation Limit	Precision QC Check	Precision Acceptance Criteria	Accuracy/Bias QC Check	Accuracy/Bias Acceptance Criteria	Corrective Action (CA)	Person Responsible for (CA)
pH SW-846 9040B	0.2 pH units	Replicate Measurements	RPD < 20%	Calibration with pH buffer solutions (4 or 10, plus 7)	Slope between 90-102	1.Check with new buffer 2.Repair/replace meter 3.Recalibrate	Field Services Manager or Field Team Member
Conductivity SW-846 9050A	0.1umho/cm	Replicate Measurements	RPD < 20%	Calibration with KCL standard	± 5% of standard	1.Evaluate 2.Recalibrate	Field Services Manager or Field Team Member
Temperature EPA 170.1	0.1°c	Replicate Measurements	RPD < 20%	Calibration against pH meter temp probe	± 0.1°c	1.Recalibrate 2.Replace ther-momter	Field Services Manager or Field Team Member
Dissolved Oxygen SM4500-OC	200 ug/l	Replicate Measurements	RPD < 20%	Calibration with standard solution	Per manufacturer operation manual	1.Evaluate 2.Recalibrate	Field Services Manager or Field Team Member
PID Organics (Air Monitoring)	Variable, depending on compound, and field conditions	Replicate Measurements	RPD < 20%	Initial calibration with zero and span gas (isobutylene)	± 5% of span gas	Recalibrate	Field Services Manager or Field Team Member

Table 4-2
Analytical Quality Control Check Samples

Type	QC Check	Frequency	Data Quality Indicator (DQI)	Acceptance Criteria	Corrective Action (CA)	Person Responsible for (CA)
Field	Field Duplicate	1 per 20 sample per matrix	Precision	RPD< 50% (Soil) RPD = 30% water	N/A	N/A
Field	Equipment Blank	1 per sampling device per day	Accuracy/ Bias	<PQL	Evaluate results for field samples	Project Chemist
Field	Trip Blank	1 per cooler (VOAs)	Accuracy/ Bias	<PQL	Evaluate results for field samples	Project Chemist
Field	Temperature Blank	1 per cooler	Accuracy/ Bias	4°c ± 2°c	1. Contact PM 2. Evaluate need for resampling	Project Chemist
Laboratory-Organics	Surrogates	Samples, Spikes, and blank	Accuracy/ Bias	Appendices B and C	Reanalyze, evaluate for interference	Analyst
Laboratory-Organics & Inorganics	Matrix Spike Matrix Spike Duplicate	1 per 20 sample batch/matrix 1 per 20 sample batch/matrix	Accuracy/ Bias Precision	Appendices B and C	Evaluate based on LCS, other QC results, narrate	Analyst
Laboratory-Organics & Inorganics	Method Blank	1 per 20 sample batch/matrix	Accuracy/ Bias	<PQL	1. Re-prep and re-analyze blank samples if necessary 2. Narrate	Analyst
Laboratory-Organics & Inorganics	Laboratory Control Sample	1 per 20 sample batch/matrix	Sensitivity	Appendices B and C	Reanalyze sample; evaluate; narrate	Analyst
Laboratory-Organics	Internal Standards	Samples, spikes, and blank	Sensitivity	Appendices B and C	Reanalyze sample; evaluate; narrate	Analyst
Laboratory Inorganics	Serial Dilutions (ICP)	Each batch of 20 samples similar matrix	Precision	± 10% of original determination	Qualify data	Analyst

Table 4-2
Analytical Quality Control Check Samples

Type	QC Check	Frequency	Data Quality Indicator (DQI)	Acceptance Criteria	Corrective Action (CA)	Person Responsible for (CA)
Laboratory Inorganics	Interference Check sample (ICP)	Each wavelength after ICV at beginning of run	Precision	± 20% of the true value for the analytes	Recalibrate Instrument	Analyst
Laboratory Inorganics	Post Digestion Spike	When Matrix Spikes are outside acceptance windows	Accuracy/Bias	AA 85% - 115% ICP 75% - 125%	Quality data	Analyst
Laboratory Inorganics	Blank - ICB	After every calibration/ verification	Accuracy/Bias	<PQL	Correct problem, recalibrate, and re-analyze	Analyst
Laboratory Inorganics	Blank - CCB	After every calibration/ verification	Accuracy/Bias	<PQL	Correct problem, recalibrate, and re-analyze	Analyst
Laboratory Inorganics	Blank - Prep	1 per 20 sample batch/matrix	Accuracy/Bias	<PQL	1. Re-prep and reanalyze blank and sample if necessary 2. Narrate	Analyst
Laboratory Organics & Inorganics	Initial Calibration	Per SOP for Method	Precision	Per SOP for Method	1. Evaluate 2. Recalibrate when QC criteria is not met	Analyst
Laboratory Organics & Inorganics	Continuing Calibration Verification	Per SOP for Method	Precision	Per SOP for Method	1. Evaluate 2. Clean system 3. Reanalyze calibration verification and associated samples and/or recalibrate	Analyst

Table 6-1
Summary of Holding Times and Preservation Requirements

Test	Test Method	Matrix	Container	Volume	Holding Time	Preservation[1]
Volatile Organic Compounds	SW-846 Method 8260B	Soil, Dry Sediment	E	3 x 5gram samples plus 125g glass bottle for % solids and effervescence determinations	48 hours to preserve, total of 14 days to analyze	low level = add EnCore to sodium bisulfate (1gram/5ml reagent water) and cool 4°C low level (effervescent samples) = add EnCore to 5ml reagent water and freeze high level = add EnCore to 10ml Methanol, and cool 4°C
		Wet Sediment	G	125 g	14 days	Cool 4°C
		Water	G	3 x 40 ml	14 days	Cool 4°C HCl to pH<2
Semivolatile Organic Compounds	SW-846 Method 8270C	Soil, Sediment	G	250 g	14 days to extract, 40 days to analyze	Cool 4°C
		Water	G (amber)	2 x 1000 ml	7 days to extract, 40 days to analyze	Cool 4°C
Metals (except Hg)	SW-846 Method 6010B/ 60202	Soil, Sediment	G	250 g	6 months	Cool 4°C
		Water	P	500 ml	6 months	Cool 4°C HNO3 to pH<2
Mercury (Hg)	SW-846 Method 7471A(soil) 7470A(water)	Soil, Sediment	G	250 g (combined with metals)	28 days	Cool 4°C
		Water	P	500 ml (combined with metals)	28 days	Cool 4°C HNO3 to pH<2
Nitroaromatics & Nitroamines	SW-846 Method 8330 (Enchem) Method 8321A (STL-Denver)	Soil, Sediment	G	1 x 100 g	14 days to extract, 40 days to analyze	Cool 4°C
		Water	G (amber)	2 x 1000 ml	7 days to extract, 40 days to analyze	Cool 4°C
% Solids	EPA 160.3	Soil, Sediment	G or P	125g	N/A	Cool, 4°C

Table 6-1
Summary of Holding Times and Preservation Requirements

Water Quality Samples

Nitrate-Nitrite	EPA 353.2	Water	G	250 ml	28 days	Cool 4°C
Sulfate	EPA 300.0	Water	P	500 ml	28 days	Cool 4°C
Chloride	EPA 300.0	Water	P	500 ml	28 days	Cool 4°C
Bromide	EPA 300.0	Water	P	500 ml	28 days	Cool 4°C

Waste Disposal Samples

TCLP Volatiles	1311ZHE 8260B	Soil	G	125g	14 days to leach 14 days to analyze	Cool 4°C
TCLP Semivolatiles Pesticides Herbicides	1311 Extraction 8270C 8081A 8151A	Soil	G	500g	14 days to leach 7 to extract 40 to analyze	Cool 4°C
TCLP Metals	1311 Extraction 6010B 7470A (Hg)	Soil	G	500g	14 days to leach 180 days after leaching to analyze (28 for Hg)	Cool 4°C

[1] All samples are to be stored at $4^{o}C \pm 2^{o}C$ unless noted otherwise. Transfer and preservation of EnCore samples will be performed by the laboratory

[2] Method 6020 will be for the analysis of As, Pb, Se, Tl. For samples with known or suspected high sulfur concentrations or other matrix issues, all metals except Hg will analyzed by 6010B.

P = Polyethylene

G = Glass

E = Encore sampler

Note: Analyses with similar container/presentation requirements may be combined as long as minimum volume requirements are met

Table 8-1

Nitroaromatic and Nitroamine Explosives by SW-846 8321A
Analyte Reporting Limits

Compound	CAS Number	Reporting Limit	Soil (ug/kg) MDL*	Reporting Limit	Water (ug/l) MDL*
HMX	2691-41-0	250	168	0.12	0.022
RDX	121-82-4	250	215	0.12	0.028
1,3,5,-Trinitrobenzene	99-35-4	250	125	0.12	0.017
1,3-Dinitrobenzene	99-65-0	250	73	0.12	0.020
Tetryl	479-45-8	250	183	0.12	0.019
2,4,6-Trinitrotoluene	118-96-7	250	111	0.12	0.049
Nitrobenzene	98-95-3	250	128	0.12	0.025
Nitroglycerin	55-63-0	5000	1090	0.12	0.049
2,4-Dinitrotoluene	121-14-2	250	116	0.12	0.016
2-Amino-4,6-dinitrotoluene	355-72-78-2	250	143	0.12	0.013
2,6-Dinitrotoluene	606-20-2	250	59	0.12	0.012
4-Amino-2,6-dinitrotoluene	1946-51-0	250	120	0.12	0.017
2-Nitrotoluene	88-72-2	250	196	0.12	0.038
4-Nitrotoluene	99-99-0	250	137	0.12	0.038
3-Nitrotoluene	99-08-1	250	152	0.12	0.019
PETN	78-11-5	2500	1010	0.12	0.020

***** - Based on current STL-Denver (January 2001) Method Detection Unit Studies. MDL values are subject to revision in accordance with laboratory MDL study updates.

Table 8-2

Nitroaromatic and Nitroamine Explosives by SW-846 8330
Analyte Reporting Limits

Compound	CAS Number	Reporting Limit	Soil (ug/kg) MDL*	Reporting Limit	Water (ug/l) MDL*
HMX	2691-41-0	200	39.71	0.40	0.0507
RDX	121-82-4	200	21.67	0.40	0.0807
1,3,5,-Trinitrobenzene	99-35-4	200	25.28	0.40	0.0605
1,3-Dinitrobenzene	99-65-0	200	19.31	0.40	0.0648
Tetryl	479-45-8	200	112.72	0.40	0.0908
2,4,6-Trinitrotoluene	118-96-7	200	38.40	0.40	0.0700
Nitrobenzene	98-95-3	200	19.68	0.40	0.0781
Nitroglycerin	55-63-0	NA	NA	NA	NA
2,4-Dinitrotoluene	121-14-2	200	20.55	0.40	0.0608
2-Amino-4,6-dinitrotoluene	355-72-78-2	200	24.41	0.40	0.0749
2,6-Dinitrotoluene	606-20-2	200	19.55	0.40	0.0449
4-Amino-2,6-dinitrotoluene	1946-51-0	200	42.19	0.40	0.0550
2-Nitrotoluene	88-72-2	200	36.28	0.40	0.0564
4-Nitrotoluene	99-99-0	200	34.23	0.40	0.0586
3-Nitrotoluene	99-08-1	200	16.88	0.40	0.0719
PETN	78-11-5	NA	NA	NA	NA

***** - Based on current Enchem-Madison (June 2001) Method Detection Unit Studies. MDL values are subject to revision in accordance with laboratory MDL study updates.

NA - Not analyzed during MDL study

Table 8-3

Appendix IX Volatile Organics by SW-846 8260B
Analyte Reporting Limits

Compound	Soil (ug/kg)		Water (ug/l)	
	Reporting Limit	MDL	Reporting Limit	MDL
Acetone	20.0	3.42	10.0	1.88
Acetonitrile	100.0	16.80	20.0	2.59
Acrolein	100.0	38.18	20.0	4.71
Acrylonitrile	100.0	5.91	20.0	2.39
Benzene	5.0	0.50	1.0	0.21
Bromodichloromethane	5.0	0.50	1.0	0.22
Bromoform	5.0	0.50	1.0	0.32
Bromomethane	10.0	0.50	2.0	0.30
2-Butanone (MEK)	20.0	2.34	5.0	0.93
Carbon disulfide	5.0	0.52	1.0	0.19
Carbon tetrachloride	5.0	0.54	1.0	0.19
Chlorobenzene	5.0	1.01	1.0	0.30
Chloroprene	5.0	0.83	1.0	0.22
Dibromochloromethane	5.0	0.50	1.0	0.38
Chloroethane	10.0	0.50	2.0	0.25
Chloroform	5.0	0.50	1.0	0.23
Chloromethane	10.0	0.91	2.0	0.30
Allyl chloride	10.0	0.54	2.0	0.20
1,2-Dibromo-3-chloropropane (DBCP)	10.0	0.68	2.0	0.25
1,2-Dibromoethane (EDB)	5.0	0.50	1.0	0.36
Dibromomethane	5.0	0.50	1.0	0.44
trans-l,4-Dichloro-2-butene	5.0	1.03	1.0	0.60
Dichlorodifluoromethane	10.0	0.62	2.0	0.23
1,1-Dichloroethane	5.0	0.65	1.0	0.17
1,2-Dichloroethane	5.0	0.56	1.0	0.28
cis-l,2-Dichloroethene	2.5	0.56	1.0	0.26
trans-l,2-Dichloroethene	2.5	0.77	0.5	0.27
1,1-Dichloroethene	5.0	0.71	1.0	0.20
1,2-Dichloropropane	5.0	0.50	1.0	0.21
cis-l,3-Dichloropropene	5.0	0.72	1.0	0.28
trans-1,3-Dichloropropene	5.0	0.53	1.0	0.42
1,4-Dioxane	500.0	42.82	200.0	16.74
Ethylbenzene	5.0	1.14	1.0	0.28
Ethyl methacrylate	5.0	0.60	1.0	0.25
2-Hexanone	20.0	1.66	5.0	0.70
Iodomethane	5.0	0.50	1.0	0.23
Isobutyl alcohol	200.0	11.66	50.0	11.08
Methacrylonitrile	50.0	5.00	10.0	1.60
Methylene chloride	5.0	0.50	1.0	0.89
Methyl methacrylate	5.0	1.29	1.0	0.30
4-Methyl-2-pentanone	20.0	1.20	5.0	0.79
Propionitrile	20.0	6.26	5.0	2.22
Styrene	5.0	1.25	1.0	0.27
1, 1, 1,2-Tetrachloroethane	5.0	1.30	1.0	0.22
1,1,2,2-Tetrachloroethane	5.0	0.50	1.0	0.31

	Soil (ug/kg)	Water (ug/l)

Table 8-3

Appendix IX Volatile Organics by SW-846 8260B

Compound	Reporting Limit	MDL	Reporting Limit	MDL
Tetrachloroethene	5.0	1.02	1.0	0.36
Toluene	5.0	0.81	1.0	0.29
1,1,1-Trichloroethane	5.0	0.50	1.0	0.26
1,1,2-Trichloroethane	5.0	0.98	1.0	0.39
Trichloroethene	5.0	0.62	1.0	0.22
Trichlorofluoromethane	10.0	0.55	2.0	0.28
1,2,3-Trichloropropane	5.0	1.13	1.0	0.29
Vinyl acetate	10.0	4.38	2.0	0.31
Vinyl chloride	10.0	0.78	1.0	0.21
Xylenes (total)	5.0	3.08	2.0	0.95

Note : MDL values are subject to revision in accordance with laboratory MDL study updates.

Table 8-4

Appendix IX Semi Volatile Organics by Method 8270C
Analyte Reporting Limits

Compound	Soil (ug/kg)		Water (ug/l)	
	Reporting Limit	MDL	Reporting Limit	MDL
a,a-Dimethylphenethylamine	1600	174.0	50.0	35.8
Acenaphthene	330	46.0	10.0	1.0
Acenaphthylene	330	34.0	10.0	1.0
Acetophenone	330	33.0	100.0	1.4
2-Acetylaminofluorene	3300	33.0	50.0	1.0
4-Aminobiphenyl	1600	81.8	10.0	12.0
Aniline	330	57.0	10.0	6.0
Anthracene	330	78.0	10.0	1.2
Aramite	660	66.0	20.0	2.0
Benzo(a)anthracene	330	39.0	10.0	1.3
Benzo(b)fluoranthene	330	100.0	10.0	2.2
Benzo(k)fluoranthene	330	93.0	10.0	2.2
Benzo(ghi)perylene	330	70.0	10.0	1.1
Benzo{a)pyrene	330	94.0	10.0	1.9
Benzyl alcohol	330	77.0	10.0	3.0
bis(2-Chloroethoxy)methane	330	74.0	10.0	1.4
bis(2-Chloroethyl) ether	330	49.0	10.0	1.8
bis(2-Chloroisopropyl) ether	330	69.0	10.0	1.3
bis(2-Ethylhexyl) phthalate	330	69.0	10.0	1.9
4-Bromophenyl phenyl ether	330	71.0	10.0	1.3
Butyl benzyl phthalate	330	34.0	10.0	1.9
4-Chloroaniline	330	47.0	10.0	7.3
Chlorobenzilate	330	36.5	10.0	1.8
4-Chloro-3-methylphenol	330	95.0	10.0	1.3
2-Chloronaphthalene	330	33.0	10.0	1.4
2-Chlorophenol	330	38.0	10.0	1.5
4-Chlorophenyl phenyl ether	330	71.0	10.0	1.5
Chrysene	330	53.2	10.0	1.5
Diallate	660	66.0	20.0	2.0
Dibenz(a,h)anthracene	330	47.0	10.0	1.6
Dibenzofuran	330	82.0	10.0	1.3
Di-n-butyl phthalate	330	76.0	10.0	2.1
1,2-Dichlorobenzene	330	64.0	10.0	1.9
1,3-Dichlorobenzene	330	71.0	10.0	2.5
1,4-Dichlorobenzene	330	55.0	10.0	2.2
3,3.-Dichlorobenzidine	1600	70.0	50.0	16.0
2,4-Dichlorophenol	330	88.0	10.0	1.7
2,6-Dichlorophenol	330	33.0	10.0	1.0
Diethylphthalate	660	53.0	10.0	2.0
Dimethoate	660	33.5	20.0	1.1
4-Dimethylaminoazobenzene	660	42.3	20.0	2.8
7,12-Dimethylbenz(a)anthracene	660	37.8	20.0	1.0
3,3'-Dimethylbenzidine	660	57.0	20.0	8.3
2,4-Dimethylphenol	330	174.0	10.0	2.1
Dimethyl phthalate	330	85.0	10.0	1.8

Table 8-4

Appendix IX Semi Volatile Organics by Method 8270C
Analyte Reporting Limits

Compound	Soil (ug/kg)		Water (ug/l)	
	Reporting Limit	MDL	Reporting Limit	MDL
1,3-Dinitrobenzene	330.0	42.4	10.0	1.0
4,6-Dinitro-2-methylphenol	1600.0	420.0	50.0	15.0
2,4-Dinitrophenol	1600.0	500.0	50.0	16.0
2,4-Dinitrotoluene	330.0	96.0	10.0	2.4
2,6-Dinitrotoluene	330.0	100.0	10.0	2.3
Di-n-octyl phthalate	330.0	36.0	10.0	2.0
Diphenylamine	330.0	100.0	10.0	2.0
Ethyl methanesulfonate	330.0	44.5	10.0	1.0
Fluoranthene	330.0	84.0	10.0	2.0
Fluorene	330.0	76.0	10.0	1.4
Hexachlorobenzene	330.0	76.0	10.0	1.5
Hexachlorobutadiene	330.0	100.0	10.0	3.3
Hexachlorocyclopentadiene	1600.0	33.0	50.0	9.1
Hexachloroethane	330.0	50.0	10.0	3.0
Hexachloropropene	3300.0	40.0	100.0	2.0
Indeno(1,2,3-cd)pyrene	330.0	48.0	10.0	1.3
Isodrin	330.0	33.0	10.0	1.1
Isophorone	330.0	68.0	10.0	1.6
Isosafrole	660.0	66.0	20.0	3.2
Methapyrilene	1600.0	56.7	50.0	16.0
3-Methylcholanthrene	660.0	33.0	20.0	2.1
Methyl methanesulfonate	330.0	36.3	10.0	1.2
2-Methylnaphthalene	330.0	59.0	10.0	1.9
2-Methylphenol	330.0	77.0	10.0	1.6
3-Methylphenol & 4-Methylphenol	330.0	74.0	10.0	1.4
Naphthalene	330.0	70.0	10.0	1.4
1,4-Naphthoquinone	1600.0	33.0	50.0	1.0
1-Naphthylamine	330.0	83.9	10.0	7.9
2-Naphthylamine	330.0	77.9	10.0	7.6
2-Nitroaniline	1600.0	80.0	50.0	1.6
3-Nitroaniline	1600.0	85.0	50.0	6.7
4-Nitroaniline	1600.0	64.0	50.0	4.4
Nitrobenzene	330.0	85.0	10.0	1.7
2-Nitrophenol	330.0	120.0	10.0	1.7
4-Nitrophenol	1600.0	95.0	50.0	7.1
Nitroquinoline-l-oxide	3300.0	42.7	100.0	15.8
N-Nitrosodi-n-butylamine	330.0	33.0	10.0	1.5
N-Nitrosodiethylamine	330.0	37.0	10.0	1.2
N-Nitrosodimethylamine	330.0	59.0	10.0	1.7
N-Nitrosodiphenylamine	330.0	72.0	10.0	5.3
N-Nitrosodi-n-propylamine	330.0	88.0	10.0	1.8
N-Nitrosomethylethylamine	330.0	45.9	10.0	1.0

Table 8-4

Appendix IX Semi Volatile Organics by Method 8270C
Analyte Reporting Limits

	Soil (ug/kg)		Water (ug/l)	
Compound	**Reporting Limit**	**MDL**	**Reporting Limit**	**MDL**
N-Nitrosomorpholine	330.0	52.7	10.0	1.5
N-Nitrosopiperidine	330.0	33.0	10.0	1.4
N-Nitrosopyrrolidine	330.0	36.6	10.0	1.1
5-Nitro-o-toluidine	660.0	58.8	20.0	3.2
Parathion	1600.0	70.0	50.0	1.3
Pentachlorobenzene	330.0	33.0	10.0	1.4
Pentachloroethane	1600.0	33.0	50.0	1.4
Pentachloronitrobenzene	1600.0	33.0	50.0	1.0
Pentachlorophenol	1600.0	370.0	50.0	7.7
Phenacetin	660.0	48.8	20.0	1.0
Phenanthrene	330.0	37.0	10.0	1.2
Phenol	330.0	71.0	10.0	1.4
4-Phenylenediamine	1600.0	839.0	100.0	30.9
Phorate	1600.0	33.0	50.0	2.0
2-Picoline	660.0	33.0	20.0	1.4
Pronamide	660.0	33.0	20.0	1.0
Pyrene	330.0	40.0	10.0	1.7
Pyridine	660.0	400.0	20.0	12.0
Safrole	1600.0	36.7	50.0	1.7
Sulfotepp	1600.0	33.0	50.0	1.0
1,2,4,5-Tetrachlorobenzene	330.0	33.0	10.0	1.9
2,3,4,6-Tetrachlorophenol	1600.0	47.9	50.0	1.2
Thionazin	1600.0	48.2	10.0	1.2
o-Toluidine	660.0	98.3	10.0	4.6
1,2,4-Trichlorobenzene	330.0	64.0	10.0	1.8
2,4,5-Trichlorophenol	330.0	75.0	10.0	1.1
2,4,6-Trichlorophenol	330.0	50.0	10.0	1.1
O,O,O-Triethyl phosphorothioate	1600.0	35.1	50.0	1.6
1,3,5-Trinitrobenzene	1600.0	48.2	50.0	1.4

Note: MDL values are subject to revision in accordance with laboratory MDL study updates.

Table 8-5
Wisconsin Regulated Volatile Organics by SW-846 8260B
Analyte Reporting Limits

COMPOUND	WATER (UG/L)	
	REPORTING LIMIT	MDL
1, 1, 1,2-Tetrachloroethane	1.0	0.22
1,1,1-Trichloroethane	1.0	0.26
1,1,2,2-Tetrachloroethane	1.0	0.31
1,1,2-Trichloroethane	1.0	0.39
1,1-Dichloroethane	1.0	0.17
1,1-Dichloroethene	1.0	0.20
1,2,3-Trichloropropane	1.0	0.29
1,2,4-Trichlorobenzene	1.0	0.20
1,2,4-Trimethylbenzene	1.0	0.22
1,2-Dibromo-3-chloropropane (DBCP)	2.0	0.25
1,2-Dibromoethane (EDB)	1.0	0.36
1,2-Dichlorobenzene	1.0	0.24
1,2-Dichloroethane	1.0	0.28
l,2-Dichloroethene (total)	1.0	0.53
1,2-Dichloropropane	1.0	0.21
1,3,5-Trimethylbenzene	1.0	0.29
1,3-Dichlorobenzene	1.0	0.26
1,3-Dichloropropane	1.0	0.26
1,4-Dichlorobenzene	1.0	0.24
2-Butanone (MEK)	5.0	0.93
4-Methyl-2-pentanone	5.0	0.79
Acetone	10.0	1.90
Benzene	1.0	0.21
Bromodichloromethane	1.0	0.22
Bromoform	1.0	0.32
Bromomethane	2.0	0.30
Carbon disulfide	1.0	0.19
Carbon tetrachloride	1.0	0.19
Chlorobenzene	1.0	0.30
Chloroethane	2.0	0.25
Chloroform	1.0	0.23
Chloromethane	2.0	0.30
Dibromochloromethane	1.0	0.38
Dichlorodifluoromethane	2.0	0.23
Ethylbenzene	1.0	0.28
Hexane	1.0	0.25
Methyl tert-butyl ether	5.0	0.21
Methylene chloride	1.0	0.89
Naphthalene	1.0	0.15
Styrene	1.0	0.27
Tetrachloroethene	1.0	0.36
Toluene	1.0	0.29
Trichloroethene	1.0	0.22
Trichlorofluoromethane	2.0	0.28
Vinyl chloride	1.0	0.21
Xylenes	2.0	0.95

Table 8-6

Appendix IX Metals
Analyte Reporting Limits

Compound	Soil (mg/kg)		Water (ug/l)	
	Reporting Limit	MDL	Reporting Limit	MDL
Beryllium, SW846 6010B	0.5	0.029	5.0	0.22
Copper, SW846 6010B	2.0	0.091	10	0.83
Nickel, SW846 6010B	4.0	0.11	40	0.96
Vanadium, SW846 6010B	1.0	0.60	10	0.67
Zinc, SW846 6010B	2.0	0.14	20	6.6
Antimony, SW846 6010B	1.0	0.51	10	3.1
Cadmium, SW846 6010B	0.5	0.033	5	0.29
Cobalt, SW846 6010B	1.0	0.067	10	0.34
Silver, SW846 6010B	1.0	0.071	10	0.62
Tin, SW846 6010B	10.0	0.17	100	3.3
Barium, SW846 6010B	1.0	0.088	10	0.64
Chromium, SW846 6010B	1.0	0.035	10	0.56
Arsenic, SW846 6020	0.5	0.051	5.0	0.19
Lead, SW846 6020	0.1	0.021	1.0	0.20
Selenium, SW846 6020	0.5	0.062	5.0	0.15
Thallium, SW846 6020	0.1	0.002	1.0	0.020
Mercury, SW846 7471B	0.033	0.0026	0.2	0.030

Water Quality Metals
Analyte Reporting Limits

Compound	Soil (ug/kg)		Water (ug/l)	
	Reporting Limit	MDL	Reporting Limit	MDL
Iron, SW846 6010B	NA	NA	100	5.1
Sodium, SW846 6010B	NA	NA	1.0	0.22
Calcium, SW846 6010B	NA	NA	200	29
Manganese, SW846 6010B	NA	NA	10	0.38
Potassium, SW846 6010B	NA	NA	3000	500

Note: MDL values are subject to revision in accordance with laboratory MDL study updates. 6010B may be substituted for 6020 for the analysis of samples with high sulfur concentrations or matrix issues

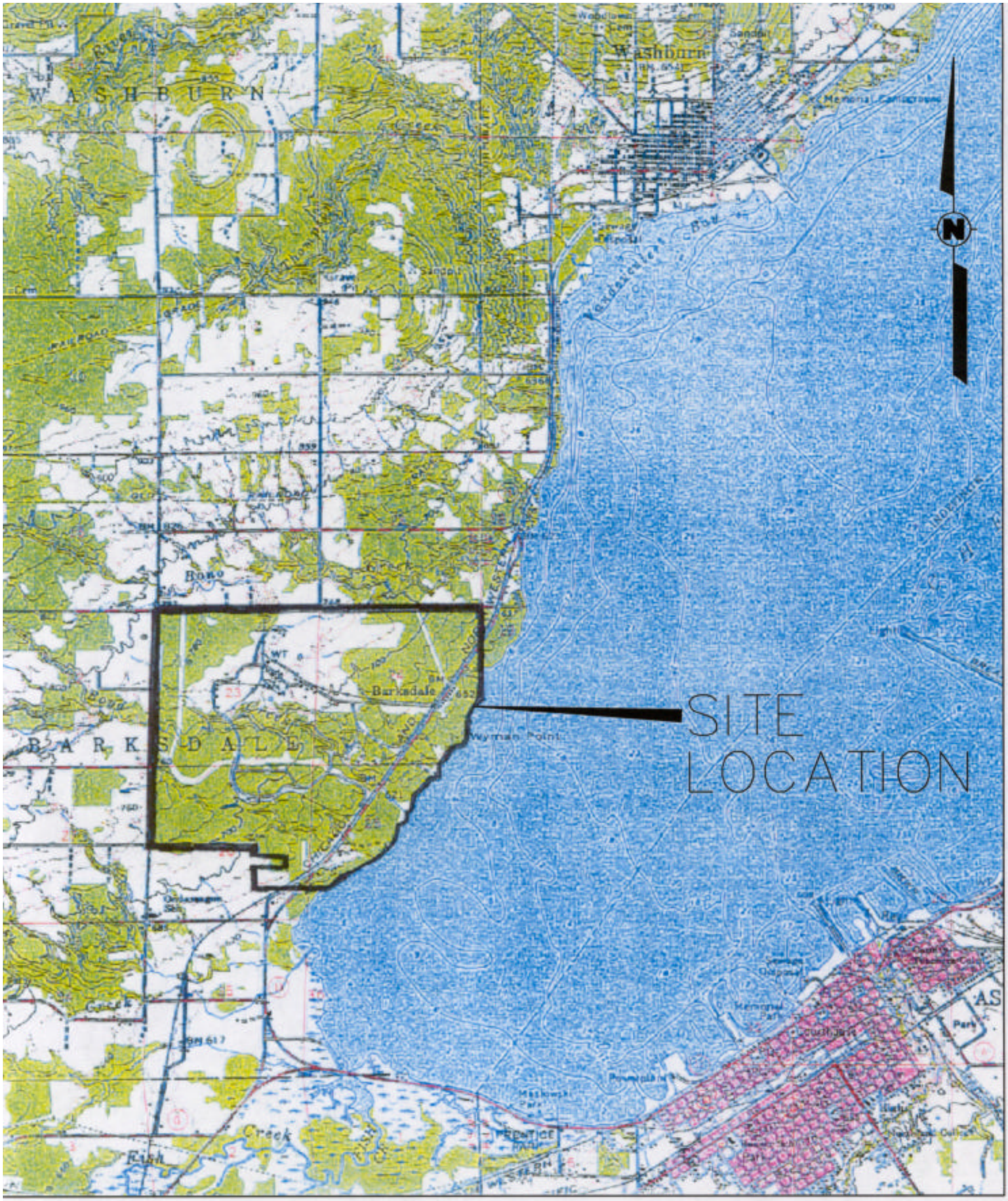
Table 8-7
Water Quality Inorganics
Analyte Reporting Limits

COMPOUND	WATER (UG/L)	
	REPORTING LIMIT	MDL
Nitrate-Nitrite, EPA 353.2	0.10	0.21
Sulfate, EPA 300.0	1.0	0.10
Chloride, EPA 300.0	1.0	0.02
Bromide, EPA 300.0	0.20	0.079

Table 8-8
TCLP Organics and Inorganics
Analyte Reporting Limits

COMPOUND	Leachate (MG/L)	
	REPORTING LIMIT	MDL
Volatiles, SW-846 Method 1311 ZHE / 8260B:		
Benzene	0.010	0.0021
2-Butanone	0.050	0.0093
Carbon Tetrachloride	0.010	0.0019
Chlorobenzene	0.010	0.0030
Chloroform	0.010	0.0023
1,2-Dichloroethane	0.010	0.0028
1,1-Dichloroethene	0.010	0.0020
Tetrachloroethene	0.010	0.0036
Trichloroethene	0.010	0.0022
Vinyl chloride	0.010	0.0021
Semivolatiles, SW-846 Method 1311/ 8270C:		
1,4-Dichlororobenzene	0.1	0.01045
2,4-Dinitrotoluene	0.1	0.00720
Hexachlorobenzene	0.1	0.01365
Hexachlorobutadiene	0.1	0.001125
Hexachloroethane	0.1	0.00860
2-Methylphenol	0.1	0.01590
3-Methylphenol and 4-Methylphenol	0.1	0.01635
Nitrobenzene	0.1	0.01225
Pentachlorophenol	0.25	0.02500
Pyradine	0.1	0.03020
2,4,5-Trichlorophenol	0.1	0.01225
2,4,6-Trichlorophenol	0.1	0.01135
Pesticides, SW-846 Method 1311 / 8081A:		
gamma-BHC (Lindane)	0.00050	0.00005
Chlordane (technical)	0.00500	0.00088
Endrin	0.00050	0.00006
Heptachlor	0.00050	0.00008
Heptachlor epoxide	0.00050	0.00005
Methoxychlor	0.00100	0.00009
Toxaphene	0.02000	0.00510
Herbicides, SW-846 Method 1311 / 8151A:		
2,4-D	0.04	0.0030
2,4,5-TP (Silvex)	0.01	0.0001
Metals, SW-846 Method 1311 / 6010B/7470A		
Arsenic, 6010B	0.5	0.0043
Barium, 6010B	10.0	0.00064
Cadmium, 6010B	0.1	0.00029
Chromium, 6010B	0.5	0.00056
Lead, 6010B	0.5	0.00120
Mercury, 7470A	0.002	0.00003
Selenium, 6010B	0.25	0.00450
Silver, 6010B	0.50	0.00062

FIGURES



SOURCE: U.S.G.S. BARKSDALE, WISCONSIN
QUADRANGLE 7.5 SERIES

SITE LOCATION MAP

Former Barksdale Works
Barksdale, Wisconsin

APPENDICES

STANDARD OPERATING PROCEDURE FOR COMPLETING CHAIN-OF-CUSTODY FORMS

Date: May 11, 2001

Prepared for: DuPont Remediation Group

CORPORATE REMEDIATION GROUP
An Alliance between
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1. PURPOSE

The purpose of this standard operating procedure is to establish a CRG/URSD proper chain-of-custody (COC) standard for tracking samples from the field to the laboratory. A proper COC is necessary if there is any possibility that the analytical data or conclusions based upon the analytical data will be used in litigation (SW846, Chapter 9, Section 9.2.2.7). The persons entering information on the COC are responsible for ensuring the document can withstand scrutiny during litigation.

2. GENERAL INFORMATION

The COC is a legal document/record that must include: facility name, facility address, phone numbers (primary contact and laboratory), sample identification, preservation, dates and times of collection of samples, possession, analyses, and laboratory performing the analyses.

3. PROCEDURES

3.1 Chain-of-Custody (COC)

The policy is to use either Option A or Option B as stated below.

3.1.1 Option A (Pre-printed COC originated by Laboratory Personnel)

If the regulatory agency requires initiation of the COC at the laboratory, use Option A. See Figures 1 and 1A for examples of COC Option A.

Laboratory Personnel do the following:

- ❏ Originate the pre-printed COC by relinquishing the bottles with a signature. The pre-printed COC contains the following information: header information (e.g., facility name, facility address, facility supervisor, project name), location code, matrix code, sample source (e.g., KIN-G-MW1), sample depths, sample type, volume, preservative (if applicable), quantity, bottle type, method and/or analyte.
- ❏ If the sample IDs are known at the time of bottle preparation, pre-print the location code, matrix code, and sample location (e.g., KIN-G-MW1) on the COC.
- ❏ If the sample IDs are **not** known at the time of bottle preparation, either pre-print the location code and matrix code only on the COC (e.g., KIN-G) or leave the sample ID blank.

Field Personnel do the following:

- ❏ If a sample is pre-printed on the COC but will never be collected:
  1. Cross out the sample on the COC.
  2. Date and initial next to the cross-out and give reason on the COC (e.g., well is dry).

- ❑ If an extra sample is collected that was not pre-printed on the laboratory relinquished COC, add this sample to a blank COC not the COC that was relinquished by Laboratory Personnel.
- ❑ If all of the samples listed on the laboratory relinquished COC cannot be collected in one day, use derived COC (see Section 5). (A way to avoid using derived COC is to have one well or boring location per COC.)
- ❑ If a sample is moved from one cooler to another:
    1. Add the comment "Moved to COC Y" (where Y is the unique COC number located on the top right-hand of the COC) in the date and time field on COC X next to the sample being moved.
    2. Add the sample to COC Y.
    3. Add the comment "moved sample from COC X" (where X is the unique COC number located on the top right-hand of the COC) in the margin of the COC Y next to the sample that was moved.

3.1.2 Option B (Pre-printed/Blank COC Originated in the Field)

- ❑ Laboratory Personnel issue forms along with the bottles to be used as chains-of-custody. These forms can be pre-printed or left blank.
- ❑ Field Personnel do the following:
    1. Collect the samples, write the sample ID according to the naming convention as described in Appendix A, if not already present, on the COC.
    2. Write the date and time of sample collection on the COC.
    3. Enter the remaining information on the COC [i.e., sample type, volume, preservative (if applicable), quantity, bottle type, method and/or analyte (if not already pre-printed on the COC)].
    4. Once the samples are ready to be shipped to the laboratory and all of the aforementioned information has been entered for the samples collected, relinquish the samples to the laboratory with his/her signature, date, and time (see Figure 2 and 2A for examples of Option B).

4. SIGNATURES

4.1 Option A

If Laboratory Personnel initiate the COC:

1. Laboratory Personnel relinquish the bottles with a signature.
2. Project Manager designates Field Personnel.
3. Field Personnel receive the cooler(s) from the courier (i.e., Laboratory/Federal Express/Airborne).
   At this time, Field Personnel sign the shipping paperwork. OR, if someone other than

Field Personnel is designated by Project Manager to receive the bottles from Courier, that person signs the shipping paperwork upon receipt of the coolers.

4. Field Personnel:
   - ❑ Check contents of cooler against COC
   - ❑ Sign the COC in the "Received By" box.
   - ❑ Relinquish the samples to the laboratory once they have finished sampling. (Note: If more than one person is in the field sampling, the person receiving the bottles/samples must also relinquish the bottles/samples.
5. Laboratory personnel:
   - ❑ Cross-out the unused "Received By/Relinquished By" boxes prior to signing.
   - ❑ Sign the COC upon receipt of the samples.
6. Field Personnel file and keep the Federal Express/Airborne bill of lading **to** and **from** the site (if possible).

4.2 Option B

If Laboratory Personnel did not initiate the COC:

1. Field Personnel sign the COC upon completion of sampling in the Relinquished By box.
2. Laboratory Personnel sign the COC upon receipt of the samples and cross-out the unused "Received By/Relinquished By" boxes.
3. Field Personnel file and keep the Federal Express/Airborne bill of lading **from** the site (if possible).

5. DERIVED COCS

(Necessary to complete the record of custody when using COC Option A and all of the samples on the pre-printed COC cannot be collected on a single day.)

- ❑ If the COC was originated by Laboratory Personnel with a relinquished signature and all of the samples listed on the COC cannot be collected and sent in one shipment, Field Personnel must use the Derived COCs. (One way to avoid the Derived COC is to list one well or boring location per COC.)
- ❑ Field Personnel must write the sample IDs of the samples that were collected that day and are to be shipped to the laboratory on the Derived COC. It is important that Field Personnel:
  1. Transcribe all of the information pertaining to the sample (e.g., correct sample ID, parameters, preservative, etc.) on the Derived COC.
  2. Reference the Derived COC# on the Original COC in the "Date and Time" boxes of the sample that was transcribed onto the Derived COC.
  3. If the Derived COC doesn't have a number, Field Personnel must assign a number. The assigned number may be the original COC number followed by a –1.

- The original COC remains in the field until all the samples listed on the COC have been collected.
- Field Personnel send the original COC with the last shipment of samples listed on the original COC (see Figure 3 and 3A for examples of "Derived COC").

6. "CROSS OUTS" ON COC

- If corrections are made to the COC while in the field, Field Personnel must date and initial next to the item that was crossed out.
- If corrections are to be made to the COC **after** it has left the field, ADQM Personnel:
  1. Document the error (i.e., email or send out the COC Exception Report form).
  2. Send the email/COC Exception Report form to the person requesting the correction (if other than ADQM Personnel) for signature.
- Once the requestor has reviewed the documentation, he/she sends an email acknowledging the correction or mails the COC Exception Report form back to ADQM personnel with a signature.
- ADQM keeps the original with the file and sends a copy to Laboratory Personnel and to the project manager.

7. LOCATION OF COC WITH RESPECT TO COOLER

Laboratory Personnel:

1. Print the COC on carbon paper so that all parties handling the samples can maintain a copy in their files.
2. Place the original COC or form (which will become a COC once a signature has been added) inside the cooler when shipped to the field.

Field Personnel:

1. Place the original COC and laboratory copy in the cooler containing the samples listed on that COC.
2. Keep one carbon copy of the COC for their files.

8. BOTTLE LABELS

Field Personnel must make sure that the bottle label contains the full sample ID (see Appendix A), the preservative added, the number of bottles, the analyses, and whether or not the sample is filtered. The information on the bottle label must match the information on the COC.

9. DATE/TIME OF SAMPLE COLLECTION

Field Personnel must:

1. Write the date on COC as MM/DD/YY (e.g., 8/31/99)
2. Write the time on COC in 24 hour or military time (e.g. 1330). The time of collection is recorded as the time the sample was initially taken. A separate time of collection is not required for each parameter (e.g., time for volatiles, time for semivolatiles, etc.) The date and time of collection of the matrix spike and matrix spike duplicate samples are the same date and time as the original sample.

10. CUSTODY SEALS

- Laboratory Personnel include custody seals with each cooler shipment.
- Field Personnel:
  1. Pack the samples on ice in the cooler.
  2. Once the cooler is ready for shipment, tape the custody seals to the broad side of the cooler lid opposite the hinges in such a way that the seals will be broken if the cooler is opened.
  3. Sign and date the custody seals prior to shipment to Laboratory Personnel. If Field Personnel break the seals of the cooler prior to shipment (e.g., to re-ice the samples), Field Personnel must attach another set of seals to the cooler with the Field Personnel's signature and the date.
  4. If specified in the QAPP, attach custody seals to the bottles. Place the seal over the cap of the bottle and down both sides in such a way that, if the cap is unscrewed, the seal will be broken (see Appendix B for example custody seal).

11. COOLER NUMBERS

ADQM Personnel instruct the laboratories to write cooler numbers on coolers and associated COC containing samples to be analyzed for volatiles (e.g., label attached with cooler number or cooler number written directly on cooler).

12. SPECIAL REQUESTS/CONCERNS

Field Personnel use comment section of COC for special requests/concerns such as analyze within 7 days, high PID reading, etc.

13. STEP-BY-STEP INSTRUCTIONS

All personnel can follow the step-by-step instructions on how to complete a COC (see Appendix C).

FIGURES

Figure 1 ← **Example of laboratory originated COC sent to the field**

Lancaster Laboratories

2425 New Holland Pike PO Box 12425 Lancaster, PA 17605-2425 **No. 123**

Job Number: 7035-504116-772000

Facility Name: DuPont Cape Fear	**Telephone Number:** 910-371-4409	**Method of Shipping:** Fed Ex	**Shipping Instructions:** Priority Overnight		
Facility Address: State Road 1426, Leland, NC 28451	**Comments:**				
Facility Supervisor: Bill Jones					
Process Producing Sample: Indicator GW					
Employee(s) Sampling:					
Other Employee(s) Handling:					

Sample Description	Date	Time	Sample Type	Bottle Volume (ml)	Preservative	Quantity	Bottle Type	8260B									
CAP-G-MW-30			WW	40	HCL	2	V	X									
		Laboratory relinquishes the bottles by signing and dating here															

Bottles Relinquished by Bob Adams	**Date/ Time** 03/22/01	10:00	**Bottles Received by**		**Date/ Time**		**Condition of samples upon arrival:**	
Bottles Relinquished by	**Date/ Time**		**Bottles Received by**		**Date/ Time**		**Signature:**	
Bottles Relinquished by	**Date/ Time**		**Bottles Received by**		**Date/ Time**		**Date:**	
Bottles Relinquished by	**Date/ Time**		**Bottles Received by**		**Date/ Time**		**Temp of Samples on Arrival: _____C**	

Figure 1A ← **Example of laboratory originated COC after completed in the field**

Lancaster Laboratories

2425 New Holland Pike PO Box 12425 Lancaster, PA 17605-2425

No. 123

Job Number: 7035-504116-772000

Facility Name: DuPont Cape Fear	**Telephone Number:** 910-371-4409	**Method of Shipping:** Fed Ex	**Shipping Instructions:** Priority Overnight
Facility Address: State Road 1426, Leland, NC 28451	**Comments:**		
Facility Supervisor: Bill Jones			
Process Producing Sample: Indicator GW			
Employee(s) Sampling: Joe Sample			
Other Employee(s) Handling: Dave White			

Sample Description	Date	Time	Sample Type	Bottle Volume (ml)	Preservative	Quantity	Bottle Type	8260B									
CAP-G-MW-30	03/22/01	1200	WW	40	HCL	2	V	X									

Laboratory personnel will accept receipt of the samples by signing and dating here.

Person who received the bottles must also relinquish the bottles by signing here

Laboratory relinquishes the bottles by signing and dating here. The point of contact receives the bottles by signing and dating here.

Bottles Relinquished by	Bob Adams	**Date/ Time** 03/21/01	10:00	**Bottles Received by**	Joe Smith	**Date/ Time** 03/22/01	900	**Condition of samples upon arrival:** Good
Bottles Relinquished by	Joe Smith	**Date/ Time** 03/22/01	1600	**Bottles Received by**	- ---------	**Date/ Time** -------	------	**Signature:** Bob Adams
Bottles Relinquished by	----------	**Date/ Time** ---------	---------	**Bottles Received by**	----------	**Date/ Time** ---------	-------	**Date:** 03/23/01
Bottles Relinquished by	---------- -	**Date/ Time** ---------	---------	**Bottles Received by**	Bob Adams	**Date/ Time** 03/23/01	930	**Temp of Samples on Arrival: __2_C**

Figure 2 ← Example of COC to be originated in the field

Lancaster Laboratories

2425 New Holland Pike PO Box 12425 Lancaster, PA 17605-2425 **No. 124**

Job Number:																		
Facility Name: DuPont Cape Fear		**Telephone Number:** 910-371-4409					**Method of Shipping:**					**Shipping Instructions:**						
Facility Address: State Road 1426, Leland, NC 28451					**Comments:**													
Facility Supervisor: Bill Jones																		
Process Producing Sample: Indicator GW																		
Employee(s) Sampling:																		
Other Employee(s) Handling:																		
Sample Description	**Date**	**Time**	**Sample Type**	**Bottle Volume (ml)**	**Preservative**	**Quantity**	**Bottle Type**	8260B										
CAP-G-MW-30			WW	40	HCL	2	V	X										
Bottles Relinquished by		**Date/ Time**			**Bottles Received by**				**Date/ Time**		**Condition of samples upon arrival:**							
Bottles Relinquished by		**Date/ Time**			**Bottles Received by**				**Date/ Time**		**Signature:**							
Bottles Relinquished by		**Date/ Time**			**Bottles Received by**				**Date/ Time**		**Date:**							
Bottles Relinquished by		**Date/ Time**			**Bottles Received by**				**Date/ Time**		**Temp of Samples on Arrival: _____C**							

Figure 2A ← **Example of COC to be originated in the field once completed in the field**

Lancaster Laboratories

2425 New Holland Pike PO Box 12425 Lancaster, PA 17605-2425

No. 124

Job Number: 7035-504116-772000

Facility Name:	DuPont Cape Fear	**Telephone Number:**	910-371-4409	**Method of Shipping:**	Federal Express
Facility Address:	State Road 1426, Leland, NC 28451	**Comments:**		**Shipping Instructions:**	Priority Overnight
Facility Supervisor:	Bill Jones				
Process Producing Sample:	Indicator GW				
Employee(s) Sampling:	Bob Adams				
Other Employee(s) Handling:	Dave White				

Sample Description	Date	Time	Sample Type	Bottle Volume (ml)	Preservative	Quantity	Bottle Type	8260B
CAP-G-MW-30	03/22/01	1030	WW	40	HCL	2	V	X

Field Personnel initiates the COC upon relinquishment of the samples by signing and dating here.

Laboratory personnel will accept receipt of the samples by signing and dating here.

Bottles Relinquished by	Bob Adams	**Date/ Time**	03/22/01	15:00	**Bottles Received by**	- ---------	**Date/ Time**	- ---------	- ------	**Condition of samples upon arrival:** Good
Bottles Relinquished by	- ---------	**Date/ Time**	- ---------	- ---------	**Bottles Received by**	- ---------	**Date/ Time**	- ---------	- ------	**Signature:** Joe Smith
Bottles Relinquished by	- ---------	**Date/ Time**	- ---------	- ---------	**Bottles Received by**	- ---------	**Date/ Time**	- ---------	- ------	**Date:** 03/23/01
Bottles Relinquished by	- ---------	**Date/ Time**	- ---------	- ---------	**Bottles Received by**	Joe Smith	**Date/ Time**	03/23/01	900	**Temp of Samples on Arrival: 3C**

Figure 3

ORIGINAL COC ← Original COC

Lancaster Laboratories

2425 New Holland Pike PO Box 12425 Lancaster, PA 17605-2425 **No. 123**

Job Number: 7035-504116-772000

Facility Name: DuPont Cape Fea	**Telephone Number:** 910-371-4409	**Method of Shipping:** Fed Ex	**Shipping Instructions:** Priority Overnight
Facility Address: State Road 1426, Leland, NC 28451	**Comments:**		
Facility Supervisor: Bill Jones			
Process Producing Sample: Indicator GW			
Employee(s) Sampling: Bob Adams			
Other Employee(s) Handling:			

Sample Description	Date	Time	Sample Type	Bottle Volume (ml)	Preservative	Quantity	Bottle Type	8260B									
CAP-G-MW-30	See COC 123-1		WW	40	HCL	2	V	X		Copy all of the information wrt CAP-G-MW-30 onto derived COC							
CAP-G-MW-28			WW	40	HCL	2	V	X									
CAP-G-MW-28D			WW	40	HCL	2	V	X									
CAP-G-MW-29			WW	40	HCL	2	V	X									

Bottles Relinquished by	Joe Smith	**Date/ Time** 03/22/01	9:00	**Bottles Received by**	**Date/ Time**	**Condition of samples upon arrival:**
Bottles Relinquished by		**Date/ Time**		**Bottles Received by**	**Date/ Time**	**Signature:**
Bottles Relinquished by		**Date/ Time**		**Bottles Received by**	**Date/ Time**	**Date:**
Bottles Relinquished by		**Date/ Time**		**Bottles Received by**	**Date/ Time**	**Temp of Samples on Arrival: _____C**

Figure 3

DERIVED COC FROM ORIGINAL COC ← Derived COC

Lancaster Laboratories

2425 New Holland Pike PO Box 12425 Lancaster, PA 17605-2425

No. 123-1

Job Number: 7035-504116-772000

Facility Name: DuPont Cape Fea	**Telephone Number:** 910-371-4409			**Method of Shipping:** Fed Ex				**Shipping Instructions:** Priority Overnight											
Facility Address: State Road 1426, Leland, NC 28451	**Comments:**																		
Facility Supervisor: Bill Jones																			
Process Producing Sample: Indicator GW																			
Employee(s) Sampling: Bob Adams																			
Other Employee(s) Handling: Dave White								8260B											
Sample Description	**Date**	**Time**	**Sample Type**	**Bottle Volume (ml)**	**Preservative**	**Quantity**	**Bottle Type**												
CAP-G-MW-30	03/23/01	1000	WW	40	HCL	2	V	X											
Field Personnel must relinquish																			
the samples by signing and dating here.																			
Bottles Relinquished by	Bob Adams	**Date/ Time**	03/23/01	15:00	**Bottles Received by**			- ---------	**Date/ Time**	- ---------	- ---------	**Condition of samples upon arrival:**						Good	
Bottles Relinquished by	- ---------	**Date/ Time**	- ---------	- ---------	**Bottles Received by**			- ---------	**Date/ Time**	- ---------	- ---------	**Signature:**		Joe Smith					
Bottles Relinquished by	- ---------	**Date/ Time**	- ---------	- ---------	**Bottles Received by**			- ---------	**Date/ Time**	- ---------	- ---------	**Date:**		03/24/01					
Bottles Relinquished by	- ---------	**Date/ Time**	- ---------	- ---------	**Bottles Received by**			Joe Smith	**Date/ Time**	03/24/01	930	**Temp of Samples on Arrival: 3 C**							

Laboratory personnel will accept the samples by signing and dating here.

Number of derived COC to tie it back to the original COC.

Add all information wrt CAP-G-MW-30 from original COC.

Figure 4
COC Exception Report Form

Job Name: _____ Site: _____
Form Initiated By/Date: _____ Date Samples Collected: _____
Responsible Party: _____

Nonconformance (check appropriate box):

Category I: Sample Collection

- ☐ 1. Sample Containers Broken in Field
- ☐ 2. Requested Measurements Not Performed
- ☐ 3. Sample Not Collected
- ☐ 4. Sample ID Incorrect

*Explanation*_____
_____

Category II: Sample Receiving

- ☐ 5. Holding Time Exceeded By_____
- ☐ 6. Test added by Client After Login (*specify*)
- ☐ 7. Sample Received Broken/Leaking
- ☐ 8. Sample Received in Improper Container
- ☐ 9. No Sample ID on Container
- ☐ 10. Sample ID Does Not Match Paperwork
- ☐ 11. Volatile Sample Received With Headspace
- ☐ 12. COC Not Completed
- ☐ 13. Sample Temperature Exceeds 4°C

Category III: Other

- ☐ 14. _____
- ☐ 15. _____

CORRECTIVE ACTION

Root Cause:
_____
_____
_____*Initials/Date*: _____

Corrective Action:
_____
_____
_____*Initials/Date*: _____

Action to Prevent Recurrence:
_____
_____
_____*Initials/Date:* _____

Manager Review:

Date Manager Aware of Problem: _____Manager's Initials: _____
Manager's Comments: _____
_____
_____
_____

APPENDICES

SAMPLE IDENTIFIER CODING

APPENDIX A

PURPOSE

Sample identifiers are used in the sampling plan, on the Chain-of-Custody, and on sample bottles to identify samples as they are created in the field. When the samples are delivered to the laboratory for analysis, the sample identifier on each sample bottle is verified on the Chain-of-Custody, and then it is entered into the laboratory's information system. When the laboratory analyses are completed, a laboratory report and an electronic deliverable are provided by the lab. The sampleid ties these results to the appropriate sample.

By encoding the sample identifier with certain pieces of information about the sample, several things are accomplished. 1)It provides a mechanism for identifying the samples uniquely. 2)It conveys information about the type and location of the sample to those who are setting up the sampling program, those that are working in the field, and those that are reviewing the data. 3)It enables the data management system to automatically decode the information and store the individual data items in separate database fields for later use in data analysis and reporting.

FORMAT

Figure 1 illustrates the sample identifier coding format. The fields making up the sample identifier are described in detail on the following page. Examples of different sample identifiers are also provided.

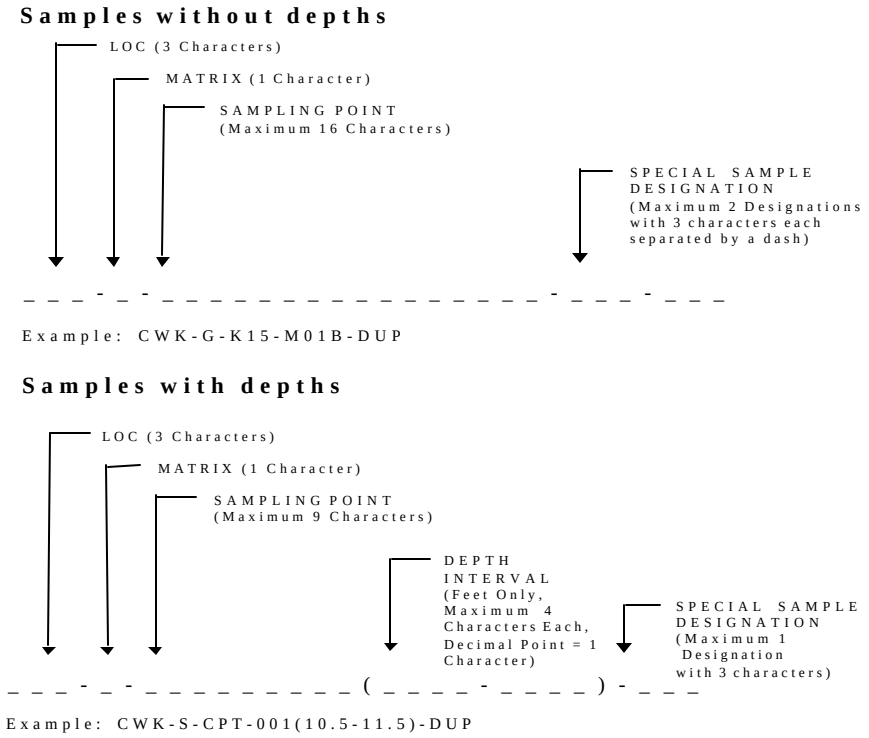


Figure 1

SAMPLE IDENTIFIER CODING

FIELDS

Sample identifiers should consist of the fields listed below concatenated in the order they are listed. The LOC, MATRIX, and SAMPLING POINT are required for all samples. The other fields are optional depending on the type of sample.

LOC -- A three character code designating the plant location or site where the sampling is being done. For example, CWK designates Chambers Works; REP designates Repauno. See Table 1 for a listing of codes. Contact the Lab Service Coordinator for locations not listed. It is required on all sample identifiers and must be the first item in the sample identifier.

SAMPLE TYPE -- A one character code designating the sample type. It is required on all sample identifiers and must immediately follow the LOC code with a dash separating it from the LOC code. If an unusual sample type is being sampled that needs to be specifically classified, contact the Lab Service Coordinator to obtain a new code.

G = Ground Water	S = Soil	A = Air
W = Surface Water	E = Sediment	N = Animal Tissue
D = Drinking Water	U = Sludge	R = Plant Tissue
K = Blank Water	P = Wipe	M = Microbiological
L = Leachate	T = Waste	X = Toxicological
		Z = Other

SAMPLING POINT NAME -- The sampling point name. This may be a well identifier, soil sample code, outfall point, etc. These identifiers should conform to the plant naming convention. Thus, if the official name of a well as defined by the plant is MW-2, then MW2, MW_2, M-2, 2, etc. should not be used. Only MW-2 should be used. Dashes are permitted within the sampling point name.

A sampling point is required in all Sample Identifiers and must immediately follow the matrix code with a dash separating it from the Matrix. For sampling points without depths, the maximum length of the sampling point is 16 characters. For sampling points with depths, the maximum length of the sampling point is 9 characters. Blanks are not permitted but may be denoted by an underscore, i.e. OUTFALL_023.

Blank samples used for QC should be identified as follows: Equipment blanks should be identified as EQBLK-#. Field blanks should be identified as FBLK-#. Trip blanks should be identified as TBLK-#. The # should be replaced with 1 to the number of the particular type of blank included in the sampling event, which may extend over several days. For example, if three trip blanks are included in a sampling event extending over two days, they would be designated TBLK-1, TBLK-2, and TBLK-3

DEPTH INTERVAL-- The depth to the top of the sampling interval followed by the depth to the bottom of the sampling interval. The depth interval must be in feet, and it must be surrounded by parentheses with the top and bottom depths separated by a dash. Each depth may have a maximum of 4 characters with a decimal point counting as one character. Thus, with two decimal places, the maximum depth is 9.99 feet; with one decimal place, the maximum depth is 99.9 feet; with no decimals, the maximum depth is 9999 feet. The depth interval is required for all soil samples.

SAMPLE IDENTIFIER CODING

SPECIAL SAMPLE DESIGNATIONS -- Designates samples used for QC purposes or requiring special handling in the field. The code must be preceded by a dash. Do not specify these if they do not apply.

DUP -- 2nd sample in a duplicate sample set
DIS -- Sample filtered in the field for dissolved metals analysis.
ACR -- This sample is for Acrolein/Acrylonitrile analysis. It requires different preservation than standard VOA samples.
MS -- Matrix Spike sample.
MSD -- Matrix Spike Duplicate sample.

EXAMPLES

1. Groundwater Monitoring Well K16-M05B primary sample at Chambers Works

   CWK-G-K16-M05B

2. Groundwater Monitoring Well K16-M05B duplicate sample at Chambers Works

   CWK-G-K16-M05B-DUP

3. Groundwater Monitoring Well K16-M05B primary sample for dissolved metals at Chambers Works

   CWK-G-K16-M05B-DIS

4. Groundwater Monitoring Well MW-44 Matrix Spike samples at Repauno

   REP-G-MW-44-MS
   REP-G-MW-44-MSD

5. Monitoring Well K16-M05B duplicate sample for dissolved metals at Chambers Works

   CWK-G-K16-M05B-DIS-DUP

6. Surface Water at Outfall 023 primary sample for Acrolein/Acrylonitrile at Niagara

   NIA-W-OUTFALL_023-ACR

7. Surface Water at Outfall 23 duplicate sample for Acrolein/Acrylonitrile at Niagara

   NIA-W-OUTFALL _023-ACR-DUP

8. Soil boring D5534 primary sample at Chambers Works taken at a depth of 6.5 feet to 7 feet

   CWK-S-D5534(6.5-7)

9. Soil Sample P123 duplicate sample at Chambers Works taken at a depth from 6 to 12 inches

   CWK-S-P123(.5-1)-DUP

10. Two Trip Blanks and an Equipment Blank for sampling at Cookson

    CKS-K-TBLK-1
    CKS-K-TBLK-2
    CKS-K-EQBLK-1

TABLE 1

LOC CODES

LOC CODES

LOC	SITE_NAME	LOC	SITE NAME
ABD	ABERDEEN,MS	FLO	FLORENCE
ABN	ABERDEEN,NC	FMN	FORT MADISON
ALB	ALBANY	GEA	GEASLIN
ACT	ALLIS CHALMERS TRUST SITE	GCC	GEON - CALVERT CITY
ANT	ANTIOCH	GLV	GEON - LOUISVILLE
ASH	ASHEPOO	GCK	GILL CREEK
AST	ASTON	GLA	GLASGOW
ATH	ATHENA	GLN	GLENOLDEN
ASC	ATHENS	GLE	GLENPOOL
BAL	BALTIMORE	GSI	GRASSELLI
BAR	BARKSDALE WORKS	GRE	GREENFIELD
BMP	BARLEY MILL PLAZA	GRB	GREENSBORO
BAY	BAYPORT	GRN	GREENWOOD
BEA	BEAUMONT	HAS	HASKELL
BEL	BELLE	HAT	HATFIELD
BET	BETHANY	HER	HERMITAGE ISLAND (NOCED)
BRK	BOERKE SITE	HOE	HOECHST-CELANESE
BRE	BREVARD	JEN	JENKS
BRI	BRIDGEPORT	JNV	JOHNSONVILLE
BUR	BURNSIDE	JON	JONESBORO
CAP	CAPE FEAR	KAN	KANSAS CITY
CAR	CARLYSS	KIN	KINSTON
CRT	CARTERET	WTL	LAKE CHARLES - CONOCO
CWK	CHAMBERS WORKS	LAK	LAKE CHARLES - VISTA
CHA	CHATTANOOGA-DUPONT	LAP	LAPORTE
CHE	CHESAPEAKE	LAV	LAVERNE
CHR	CHESTNUT RUN	LON	LONOKE
CHB	CHOCOLATE BAYOU	LOS	LOS ANGELES
CHI	CHRISTINA LABS	LOU	LOUISVILLE - DUPONT
CIN	CINCINNATI	LOV	LOUVIERS
CIR	CIRCLE RIDGE	LYN	LYNDONVILLE
CVL	CIRCLEVILLE	CDV	MACON-DOCKERY
MAM	CLEVELAND	MAN	MANATI, PUERTO RICO
CFT	CLIFTON	MAR	MARTINSVILLE
CLI	CLINTON	CAM	MAY PLANT
CKS	COOKSON	MEM	MEMPHIS - DUPONT
COO	COOPER RIVER	MIL	MILBERGER
COR	CORPUS CHRISTI	SPA	MILLIKEN
MCC	DACULA	BLA	MILLIKEN CYPRUS PLANT
DCT	DEL CITY	INM	MILLIKEN DEWEY PLANT
DEL	DELISLE	MBR	MOBERLY
DOV	DOVER	MOB	MOBILE - DUPONT
FRO	DUPONT AUTOMOTIVES PLANT	MTG	MONTAGUE
REI	DUPONT-ELSTON AVENUE	MNT	MONTGOMERY - DUPONT
EAG	EAGLE RUN	MTC	MOUNT CLEMENS
ECH	EAST CHICAGO	NAS	NASHVILLE - CONOCO
EDG	EDGEMOOR	NEC	NECCO PARK-NIAGARA FALLS
ELK	ELK GROVE	NWH	NEW HAVEN
ELR	ELK RIVER LEARNING CENTER	NEW	NEWARK
EXP	EXP STATION	NPT	NEWPORT
FAY	FAYETTEVILLE	NTN	NEWTOWN
FER	FERDULA	NIA	NIAGARA FALLS
LEW	FISHING CREEK SITE	NC	NORTH CAROLINA
FLI	FLINT	NTH	NORTHEAST

LOC CODES

OLD	OLD HICKORY	SPR	SPRUANCE SITE
PAM	PAMONA	STH	STINE HASKELL
PAR	PARLIN	TAT	TATNALL STREET
PEN	PENSACOLA	TEC	TECUMSEH
PTC	PITT CONSOL	TOL	TOLEDO
PNA	POMONA	TOW	TOWANDA
POM	POMPTON LAKES	VAL	VALLEY CENTER
PON	PONTCHARTRAIN	VIC	VICTORIA
POT	POTOMAC	WWK	WASHINGTON WORKS
REP	REPAUNO	WAY	WAYNESBORO
RTP	RESEARCH TRIANGLE PARK	WAL	WEST ALTON
ROC	ROCHESTER	WLM	WILMINGTON, PA
SAB	SABINE RIVER	WUR	WURTLAND
SAR	SARTOMER	WYS	WYSHOCK
SEA	SEAFORD		
SEN	SENECA		

LOC CODES

Modifications:

August 26, 1998: Original issued.

Jan 6, 1999 Changed MATRIX to SAMPLE TYPE. Changed G Sample Type from Groundwater to Ground Water. Changed A Sample Type from Air Sample to Air. Changed P Sample Type from Wipe Sample to Wipe.

APPENDIX B

Lancaster Laboratories
Where quality is a science.

029529
CUSTODY SEAL

DATE:_____

SIGNATURE:_____

2425 New Holland Pike, Lancasater, PA 17601-5994 (717) 656-2300

Custody Seal

DATE

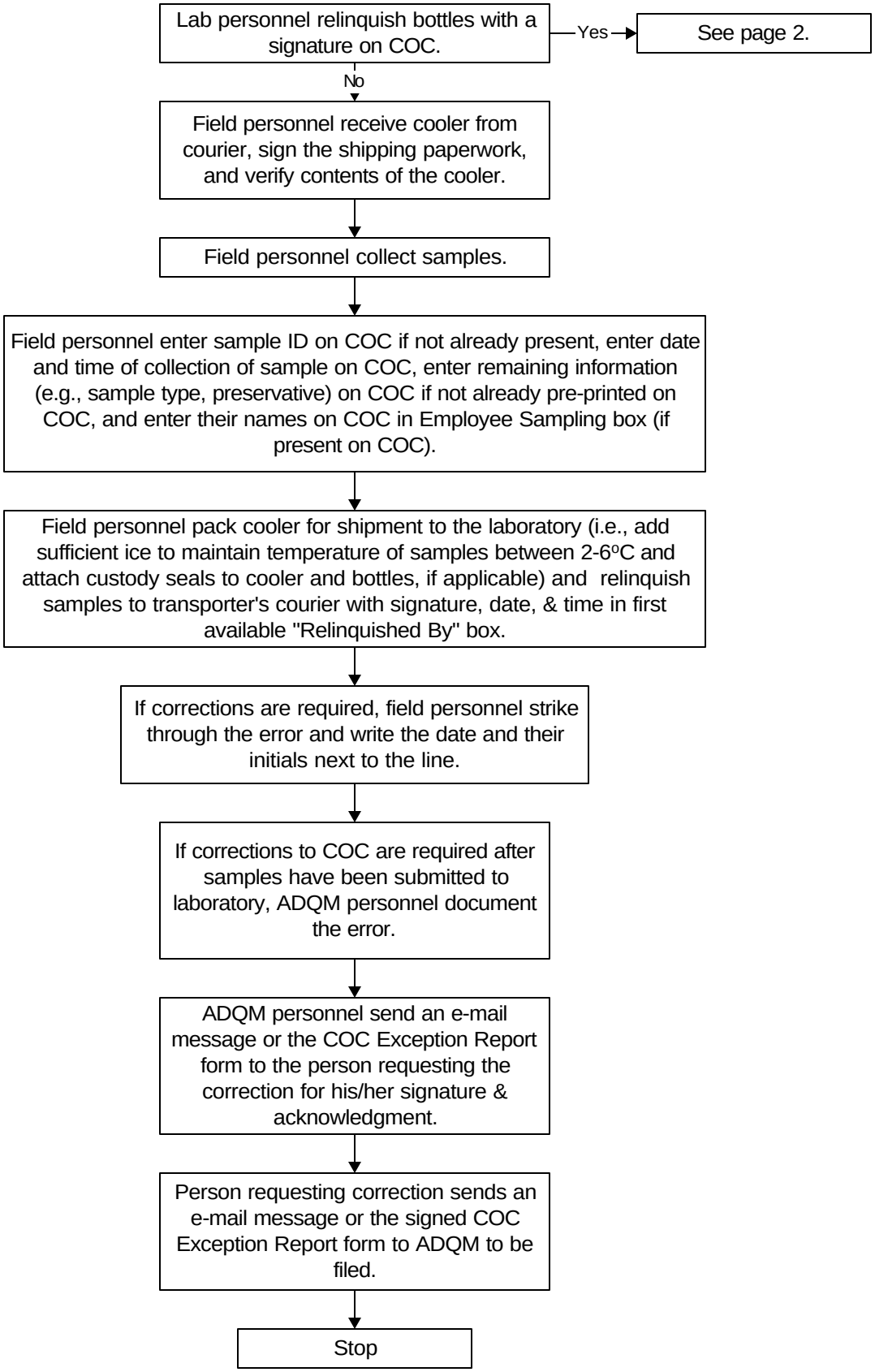
SIGNATURE

Quanterra

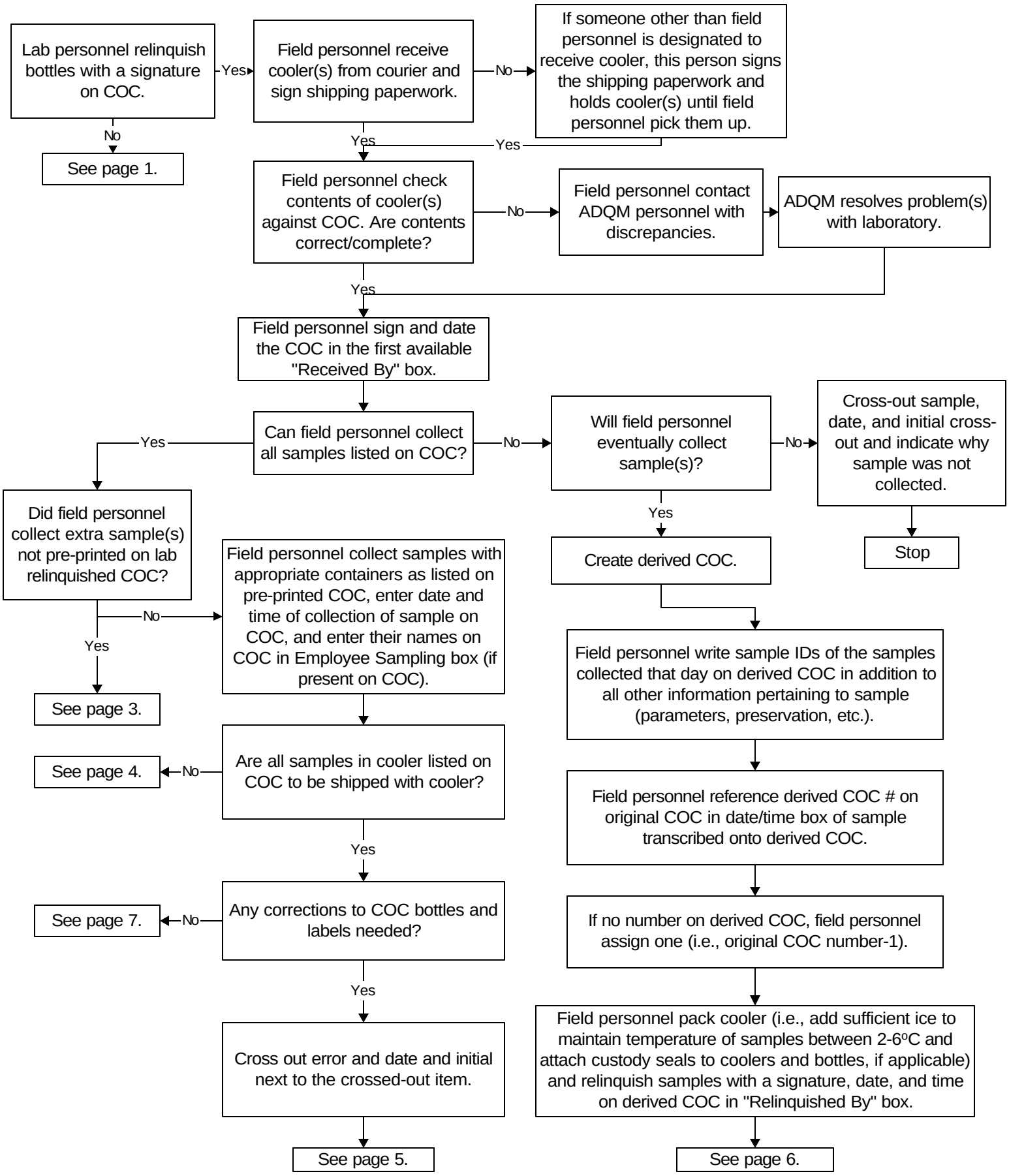
Quanterra

№

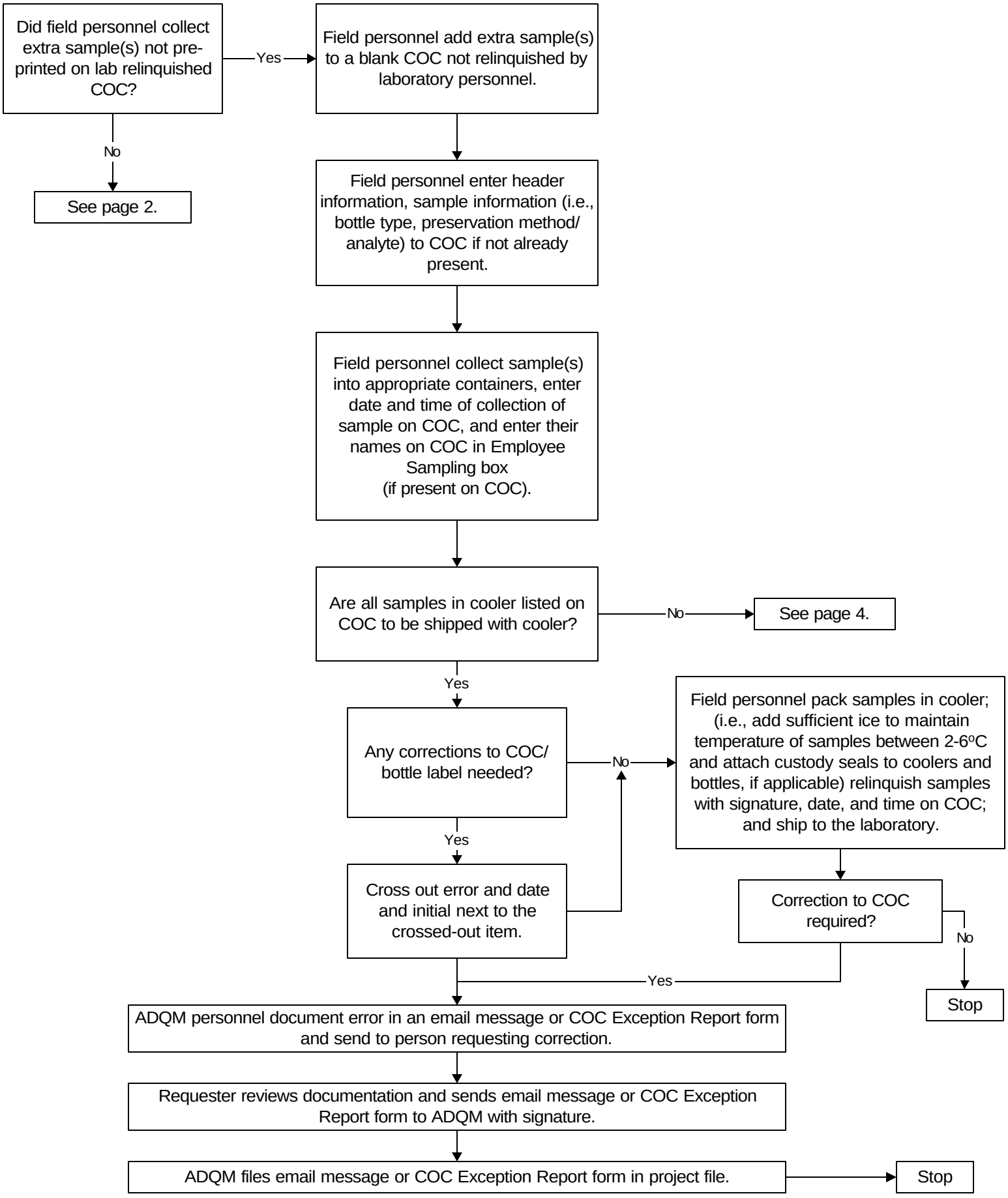
Appendix C
Chain of Custody Flow Diagram



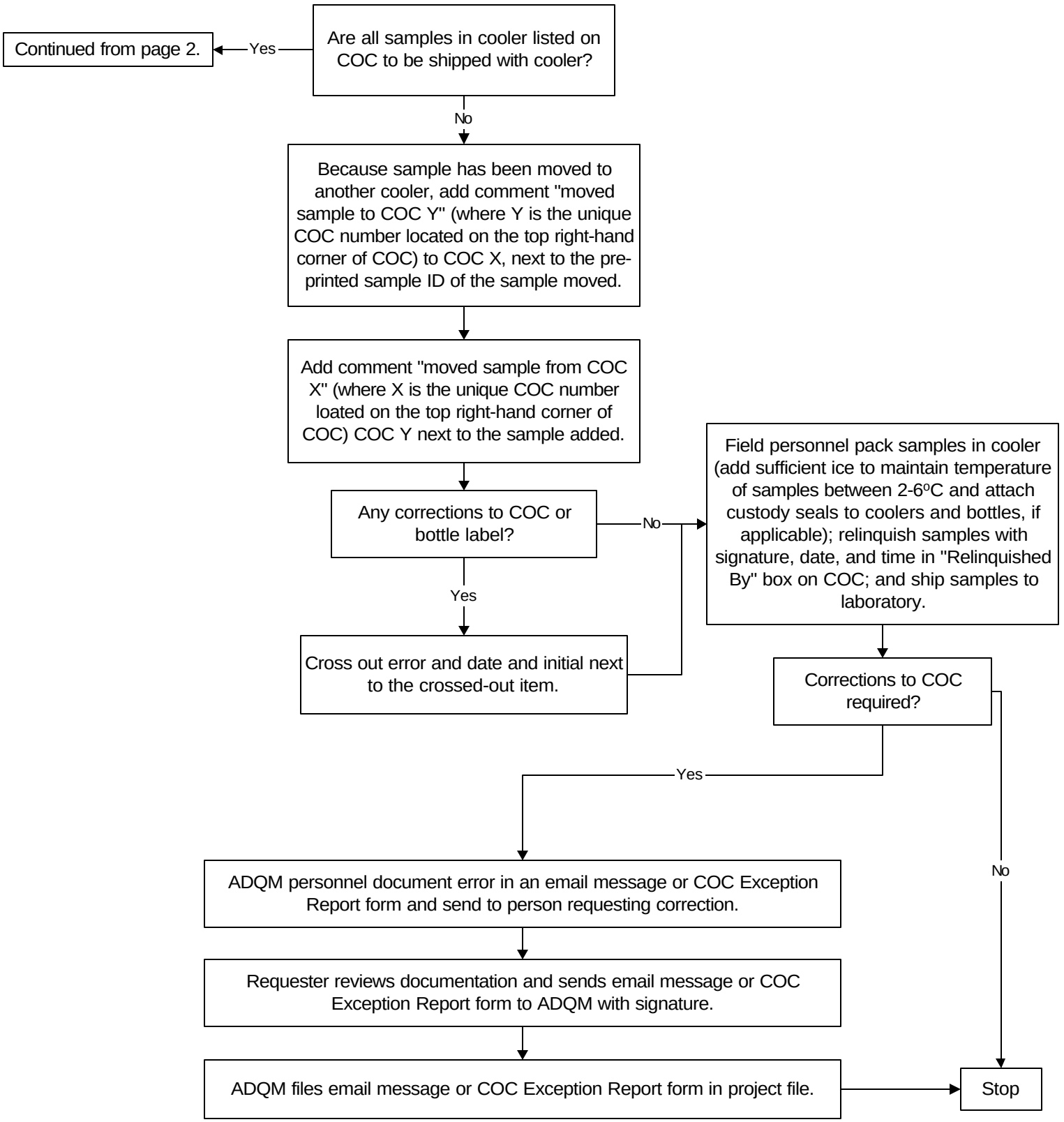
Chain of Custody Flow Diagram



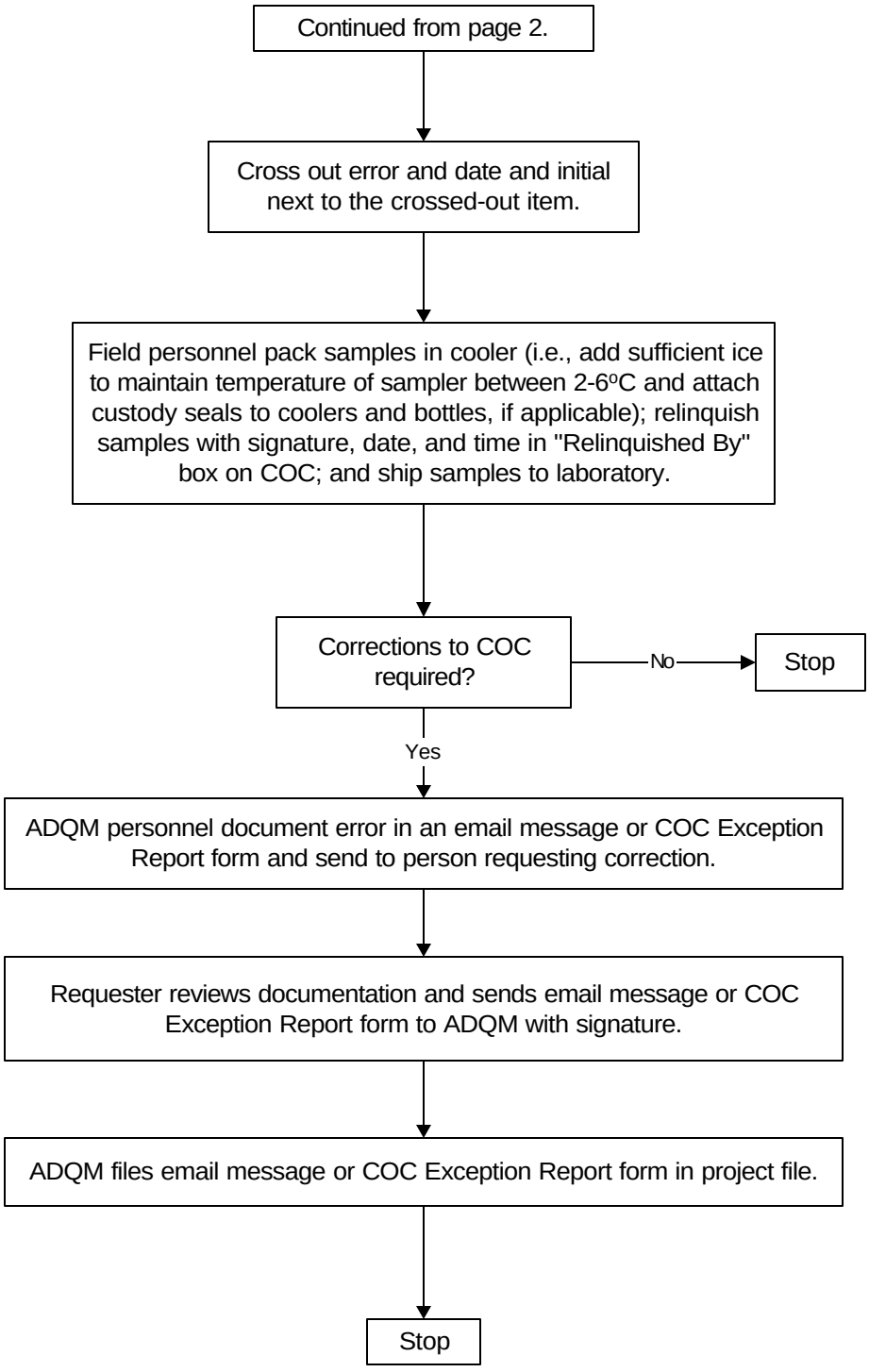
Chain of Custody Flow Diagram



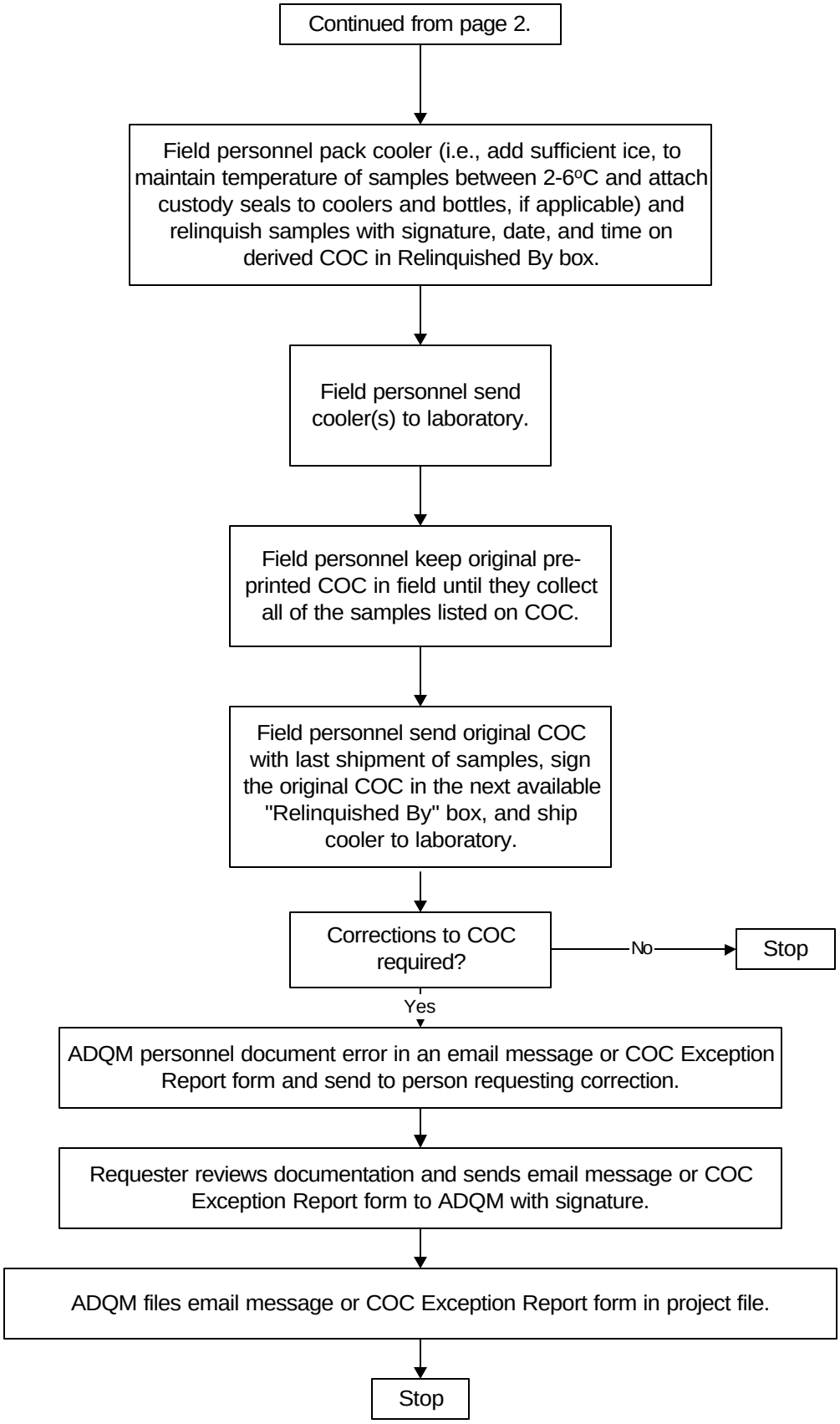
Chain of Custody Flow Diagram



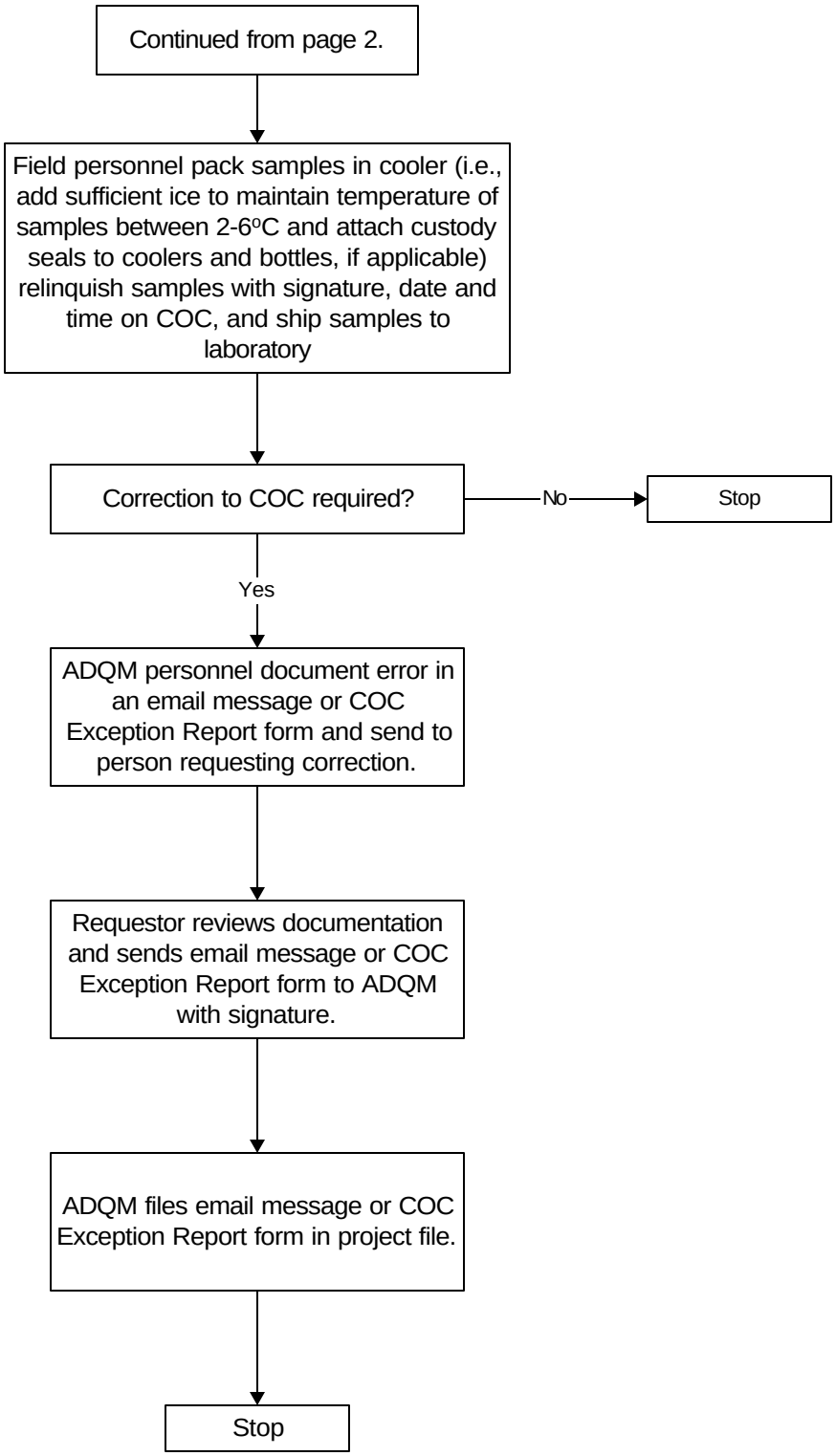
Chain of Custody Flow Diagram



Chain of Custody Flow Diagram



Chain of Custody Flow Diagram



APPENDIX B

STL-DENVER QUALITY ASSURANCE PLAN
ENCHEM QUALITY ASSURANCE PLAN

STL Reference Data Summary

Target Analyte List: DEN: AASG Explosives (Dupont)

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: . 8321A Explosives by LCMS
QC Program: AASG: LC/MS
Location: STL AASG - Denver

Target List 7189		Detection Limits			Check List 4527					Spike List 4527				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
2-Amino-4,6-dinitrotoluene	120	ug/kg	28	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	30
4-Amino-2,6-dinitrotoluene	120	ug/kg	18	ug/kg	20010502	500	ug/kg	30	120	30	600	ug/kg	30	120	30
1,3-Dinitrobenzene	120	ug/kg	22	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	90
2,4-Dinitrotoluene	120	ug/kg	34	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	30
2,6-Dinitrotoluene	120	ug/kg	12	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	30
HMX	120	ug/kg	15	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	30
Nitrobanzene	120	ug/kg	12	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	30
Nitroglycerin	120	ug/kg	60	ug/kg	20010215	500	ug/kg	30	120	30	500	ug/kg	30	120	30
3-Nitrotoluene	120	ug/kg	24	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	30
4-Nitrotoluene	120	ug/kg	75	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	30
2-Nitrotoluene	120	ug/kg	19	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	30
PETN	120	ug/kg	75	ug/kg	20010215	500	ug/kg	30	120	30	500	ug/kg	30	120	30
RDX	120	ug/kg	20	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	30
Tetryl	120	ug/kg	22	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	30
1,3,5-Trinitrobenzene	120	ug/kg	12	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	30
2,4,6-Trinitrotoluene	120	ug/kg	13	ug/kg	20010502	500	ug/kg	30	120	30	500	ug/kg	30	120	30
Nitrobenzene-d5						500	ug/kg	30	120	30	500	ug/kg	30	120	30

STL Reference Data Summary

Target Analyte List: DEN: AASG Explosives (Dupont)

Matrix: WATER
Extraction: SOLID PHASE EXTRACTION (NOMINAL)
Method:. 8321A Explosives by LCMS
QC Program: STANDARD TEST SET
Location: STL AASG - Denver

Target List 7189			Detection Limits			Check List 4395					sptko List 4395				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	units	LCL	UCL	RPD
2-Amino-4,6-dinitrotoluene	0.12	ug/L	0.013	ug/L	20010110	0.50	ug/L	35	139	30	0.50	ug/L	35	139	30
4-Amino-2,6-dinitrotoluene	0.12	ug/L	0.017	ug/L	20010110	0.50	ug/L	31	130	30	0.50	ug/L	31	130	30
1,3-Dinitrobenzene	0.12	ug/L	0.020	ug/L	20010110	0.50	ug/L	33	150	30	0.50	ug/L	33	150	30
2,4-Dinitrotoluene	0.12	ug/L	0.016	ug/L	20010110	0.50	ug/L	45	135	30	0.50	ug/L	46	135	30
2,6-Dinitrotoluene	0.12	ug/L	0.012	ug/L	20010110	0.50	ug/L	38	147	30	0.50	ug/L	38	147	30
HMX	0.12	ug/L	0.022	ug/L	20010110	0.50	ug/L	35	161	30	0.50	ug/L	35	161	30
Nitrobenzene	0.12	ug/L	0.025	ug/L	20010110	0.50	ug/L	35	125	30	0.50	ug/L	35	125	30
Nitroglycerin	0.12	ug/L	0.049	ug/L	20010110	0.50	ug/L	35	208	30	0.50	ug/L	35	208	30
3-Nitrotoluene	0.12	ug/L	0.019	ug/L	20010110	0.50	ug/L	35	132	30	0.50	ug/L	35	132	30
4-Nitrotoluene	0.12	ug/L	0.019	ug/L	20010110	0.50	ug/L	39	118	30	0.50	ug/L	39	118	30
2-Nitrotoluene	0.12	ug/L	0.019	ug/L	2001011 O	0.50	ug/L	39	118	30	0.50	ug/L	39	118	30
PETN	0.12	ug/L	0.020	ug/L	20010110	0.50	ug/L	35	190	30	0.50	ug/L	35	190	30
RDX	0.12	ug/L	0.028	ug/L	20010110	0.50	ug/L	3s	158	30	0.50	ug/L	35	158	30
Tetryl	0.12	ug/L	0.019	ug/L	20010110	0.50	ug/L	35	139	30	0.50	ug/L	35	139	30
1,3,5-Trinitrobenzene	0.12	ug/L	0.017	ug/L	20010110	0.50	ug/L	35	142	30	0.50	ug/L	35	142	30
2,4,6-Trinitrotoluene	0.12	ug/L	0.049	ug/L	20010110	0.50	ug/L	36	145	30	0.50	ug/L	35	145	30
Nitrobenzene-d5						0.50	ug/L	36	140	0	0.50	ug/L	35	140	0

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STL Reference Data Summary

Target Analyte List: DEN: EPA VOC Appendix IX List

Matrix: WATER
Extraction: PURGE AND TRAP - 25 mL purge (Waters)
Method:. Volatile Organica, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL Denver

Target List 7081		Detection Limits				Check List 4310					Spike List 4310				
Compound	RL	Units	MDL	Units	Run Dale	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acetone	10	ug/L	1.88	ug/L	20000310										
Acelonitrile	20	ug/L	2.59	ug/L	20000313										
Acrolein	20	ug/L	4.71	ug/L	20000313										
Acrylonitrile	20	ug/L	2.39	ug/L	20000313										
Benzene	1.0	ug/L	0.21	ug/L	20000313	10	ug/L	79	119	20	10	ug/L	79	119	20
Bromodichloromethane	1.0	ug/L	0.22	ug/L	20000313										
Bromofonn	1.0	ug/L	0.32	ug/L	20000313										
Bromomethane	2.0	ug/L	0.30	ug/L	20000313										
2-Butanone (MEK)	5.0	ug/L	0.93	ug/L	20000313										
tert-Butyl alcohol	50	ug/L	8.29	ug/L	20000313										
Carbon disulfide	1.0	ug/L	0.19	ug/L	20000106										
Carbon tetrachloride	1.0	ug/L	0.19	ug/L	20000314										
Chlorobenzene	1.0	ug/L	0.30	ug/L	20000313	10	ug/L	76	116	20	10	ug/L	76	116	20
Chloroprene	1.0	ug/L	0.22	ug/L	20000314										
Dibromochloromethane	1.0	ug/L	0.38	ug/L	20000313										
Chloroethane	2.0	ug/L	0.25	ug/L	20000313										
Chloroform	1 .0	ug/L	0.23	ug/L	20000313										
Chloromethane	2.0	ug/L	0.30	ug/L	20000313										
Allyl chloride	2.0	ug/L	0.20	ug/L	20600106										
1,2-Dibromo-3-chloropropane (DBCP)	2.0	ug/L	0.25	ug/L	20000313										
1,2-Dibromoethane (EDB)	1.0	ug/L	0.36	ug/L	20000314										
Dibrcmomethane	1.0	ug/L	0.44	ug/L	20000313										
trans-1,4-Dichloro-2-butene	1.0	ug/L	0.60	ug/L	20010106										
Dichlorodifluoromethane	2.0	ug/L	0.23	ug/L	20000313										
1,1-Dichloroethane	1.0	ug/L	0.17	ug/L	20000313										
1,2-Dichloroethane	1.0	ug/L	0.28	ug/L	20000313										
cis-1,2-Dichloroethene	1.0	ug/L	0.26	ug/L	20000314										
trans-1,2-Dichloroethene	0.5	ug/L	0.27	ug/L	20000314										
1,1-Dichloroethene	1.0	ug/L	0.20	ug/L	20000313	10	ug/L	79	119	20	10	ug/L	79	119	20
1,2-Dichloroethene (total)	1.0	ug/L	0.53	ug/L	20000313										
1,2-Dichloropropane	1.0	ug/L		ug/L	20000314										
cis-1,3-Dichloropropene	1.0	ug/L	0.21 0.28	ug/L	20000314										
trans-1,3-Dichloropropene	1.0	ug/L	0.42	ug/L	20000314										
1,4-Dioxane	200	ug/L	16.74	ug/L	20000313										
Ethylbenzene	1.0	ug/L	0.28	ug/L	20000313										
Ethyl methacrylate	1.0	ug/L	0.25	ug/L	20000106										
2-Hexanone	5.0	ug/L	0.70	ug/L	20000313										

Target Analyte List: DEN: EPA VOC Appendix IX List

Matrix: WATER
Extraction: PURGE AND TRAP - 25 mL purge (Waters)
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL Denver

Target List 7061	Detection Limits					Check List 4310					Spike List 4310				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Iodomethane	1.0	ug/L	0.23	ug/L	20000313										
Isobutyl alcohol	50	ug/L	11.06	ug/L	20000313										
Methacrylonitrile	10	ug/L	1.60	ug/L	20000313										
Methylene chloride	1.0	ug/L	0.89	ug/L	20600313										
Methyl methacrytate	1.0	ug/L	0.30	ug/L	20000106										
4-Methyl-2-pentanone	5.0	ug/L	0.79	ug/L	20600313										
Propionitrfle	5.0	ug/L	2.22	ug/L	20000313										
Styrene	1.0	ug/L	0.27	ug/L	20000314										
1,1,1,2-Tetrachloroethane	1.0	ug/L	0.22	ug/L	20000313										
1,1,2,2-Tetrachloroethane	1.0	ug/L	0.31	ug/L	20000313										
Tetrachloroethene	1.0	ug/L	0.36	ug/L	26000314										
Tduene	1.0	ug/L	0.29	ug/L	20000313	10	ug/L	75	122	20	10	ug/L	75	122	20
1,1,1-Trichloroethane	1.0	ug/L	0.26	ug/L	20600314										
1,1,2-Trichloroethane	1.0	ug/L	0.39	ug/L	20900314										
Trichloroethene	1.0	ug/L	0.22	ug/L	20900314	10	ug/L	81	121	20	10	ug/L	61	121	20
Trichlorofluoromethane	2.0	ug/L	0.26	ug/L	20000313										
1,2,3-Trichloropropane	1.0	ug/L	0.29	ug/L	20900313										
Vinyl acetate	2.0	ug/L	0.31	ug/L	20000106										
Vinyl chloride	1.0	ug/L	0.21	ug/L	29900313										
Xyfenes (total)	2.0	ug/L	0.95	ug/L	20000313										
4-Bromofluorobenzene						10	ug/L	79	119	0	10	ug/L	79	119	0
1,2-Dichloroethane-d4						10	ug/L	72	127	0	10	ug/L	72	127	0
Toluene-d8						10	ug/L	79	119	0	10	ug/L	79	119	0
Diiromofluoromethane						10	ug/L	60	120	0	10	ug/L	80	120	0

STL Reference Data Summary

Target Analyte List: DEN: 8270 "AP9" List (includes TCL + AP9)

Matrix: WATER
Extraction: LIQ/LIQ, CONT (A/B/N) - Acid->Base
Method; Base/Neutrals and Acids (82700)
QC Program: STANDARD TEST SET
Location: STL Denver

Target List 7066		Detection Limits			Check List 4340					Spike List 4340				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acenaphthene	10.0	ug/L	1.0	ug/L	20010124	100	ug/L	52	93	28	100	ug/L	62	93	28
Acenaphthylene	10.0	ug/L	1.0	ug/L	20010124										
Acetophenone	10.0	ug/L	1.4	ug/L	19980113										
2-Acetylaminofluorene	100	ug/L	1.0	ug/L	19980113										
4-Aminobiphenyl	50.0	ug/L	12	ug/L	19980113										
Aniline	10.0	ug/L	6.0	ug/L	20010124										
Anthracene	10.0	ug/L	1.2	ug/L	20010124										
Aramite	20.0	ug/L	2.0	ug/L	19980113										
Azobenzene	10.0	ug/L	2.0	ug/L	20010124										
Benzidine	100	ug/L	15	ug/L	19980228										
Benzo(a)anthracene	10.0	ug/L	1.3	ug/L	20010124										
Benzo(b)fluoranthene	10.0	ug/L	2.2	ug/L	20010124										
Benzo(k)fluoranthene	10.0	ug/L	2.2	ug/L	20010124										
Benzdc acid	50.0	ug/L	19	ug/L	20010124										
Benzo(ghl)perylene	10.0	ug/L	1.1	ug/L	20010124										
Benzo(a)pyrene	10.0	ug/L	1.9	ug/L	20010124										
Benzyl Jcohd	10.0	ug/L	3.0	ug/L	20010124										
bis(2-Chloroethoxy)methane	10.0	ug/L	1.4	ug/L	20010124										
bis(2-Chloroethyl) ether	10.0	ug/L	1.8	ug/L	20010124										
bis(2-Chloroisopropyl) ether	10.0	ug/L	1.3	ug/L	20010124										
bis(2-Ethylhexyl) phthalale	10.0	ug/L	1.9	ug/L	20010124										
4-Bromophenyl phenyl ether	10.0	ug/L	1.3	ug/L	20010124										
Butyl benzyl phthafate	10.0	ug/L	1.9	ug/L	20010124										
Carbazole	10.0	ug/L	1.7	ug/L	20010124										
4-Chloroaniline	10	ug/L	7.3	ug/L	20010124										
Chlorobenzilate	10.0	ug/L	1.8	ug/L	19960113										
4-Chloro-3-methylphenol	10.0	ug/L	1.3	ug/L	20010124	150	ug/L	55	95	34	150	ug/L	55	95	34
1-Chloronaphthalene	50.0	ug/L	1.3	ug/L	19960113										
2-Chloronaphthalene	10.0	ug/L	1.4	ug/L	20010124										
2-Chlorophenol	10.0	ug/L	1.5	ug/L	20010124	150	ug/L	52	92	33	150	ug/L	52	92	33
4Ehlorophenyl phenyl ether	10.0	ug/L	1.5	ug/L	20010124										
Chrysene	10.0	ug/L	1.5	ug/L	20010124										
Diaflate	20.0	ug/L	2.0	ug/L	19980113										
Dibenz(a,j)acridine	20.0	ug/L	4.1	ug/L	19980113										
Dibenz(a,h)anthracene	10.0	ug/L	1.6	ug/L	20010124										
Dibenzofuran	10.0	ug/L	1.3	ug/L	20010124										
Di-n-butyl phthalate	10.0	ug/L	2.1	ug/L	20010124										

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Target Analyte List: DEN: 8270 "AP9" List (includes TCL + AP9)

Matrix: WATER
Extraction: LIQ/LIQ, CONT (A/B/N) - Acid->Base
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL Denver

Target List 7086		Detection Limits			Check List 4340					Spike List 4340				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
1,2-Dichlorobenzene	10.0	ug/L	1.9	ug/L	20010124										
1,3-Dichlorobenzene	10.0	ug/L	2.5	ug/L	20010124										
1,4-Dichlorobenzene	10.0	ug/L	2.2	ug/L	20010124	100	ug/L	38	87	37	100	ug/L	38	87	37
3,3'-Dichlorobenzidine	50.0	ug/L	16	ug/L	20010124										
2,4-Dichlorophenol	10.0	ug/L	1.7	ug/L	20010124										
2,6-Dichlorophenol	10.0	ug/L	1.0	ug/L	19981002										
Diethyl phthalate	10.0	ug/L	2.0	ug/L	20010124										
Dimethoate	20.0	ug/L	1.1	ug/L	19980113										
p-Dimethyiamlnoazobenzene			2.8	ug/L	19980113										
7,12-Dimethylbenz(a)anthracene	20	ug/L	1	ug/L	19980113										
3,3'-Dimethylbenzidine	20.0	ug/L	8.34	ug/L	20000219										
alpha,alpha-Dimethylphenethylamine	60.0	ug/L	35.8	ug/L	20000512										
2,4-Dimethylphenol	10.0	ug/L	2.1	ug/L	20010124										
Dimethyl phthalate	10.0	ug/L	1.6	ug/L	20010124										
1,3-Dinitrobenzene	10.0	ug/L	1.o	ug/L	19980113										
1,4-Dinitrobenzene	10.0	ug/L	1.0	ug/L	19980113										
4,6-Dinitro-2-methylphenol	50.0	ug/L	15	ug/L	20010124										
2,4-Dinitrophenol	50.0	ug/L	16	ug/L	20010124										
2,4-Dinitrotoluene	10.0	ug/L	2.4	ug/L	20010124	100	ug/L	56	107	30	loo	ug/L	56	107	30
2,6-Dinitrotoluene	10.0	ug/L	2.3	ug/L	20010124										
2-sec-Butyl-4,6-dinitrophenol			2.23	ug/L	19980113										
Di-n-octyl phthalate	10.0	ug/L	2.0	ug/L	20010124										
Disulfoton	50.0	ug/L	3.3	ug/L	19980113										
Ethyl methanesulfonate	10.0	ug/L	1.0	ug/L	19980113										
Fluoranthene	10.0	ug/L	2.0	ug/L	20010124										
Fluorene	10.0	ug/L	1.4	ug/L	20010124										
Hexachlorobenzene	10.0	ug/L	1.5	ug/L	20010124										
Hexachlorobutadiene	10.0	ug/L	3.3	ug/L	20010124										
Hexachlorocyclopentadiene	50.0	ug/L	9.1	ug/L	20010124										
Hexachloroethane	10.0	ug/L	3.0	ug/L	20010124										
Hexachloropropene	100	ug/L	2.0	ug/L	19960113										
Indene	100	ug/L	1.6	ug/L	20010124										
Indeno(1,2,3-cd)pyrene	10.0	ug/L	1.3	ug/L	20010124										
Isodrin	10.0	ug/L	1.1	ug/L	19980113										
Isophorone	10.0	ug/L	1.6	ug/L	20010124										
Isosafrde	20.0	ug/L	3.2	ug/L	19980113										
Methapyrilene	50.0	ug/L	16.05	ug/L	20000512										
3-Methylcholanthrene	20.0	ug/L	2.1	ug/L	19980113										
Methyl methanesulfonate	1 a.0	ug/L	1.2	ug/L	19980113										
2-Methylnaphthalene	10.0	ug/L	1.9	ug/L	20010124										

Target Analyte List: DEN: 8270 "AP9" List (includes TCL + AP9)

Matrix: WATER
Extraction: LIQ/LIQ, CONT (A/Et/N) - Acid->Base
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL Denver

Target Ltst 7066		Detection Limits				Check List 4340					Spike List 4340				
Compound	RL	Units	MDL	Units	Run Date	Amt	Unfs	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
1-Methylnaphthalene	10.0	ug/L	1.1	ug/L	20010124										
Methyl parathion	50.0	ug/L	1.6	ug/L	19980113										
2-Methylphenol	10.0	ug/L	1.6	ug/L	20010124										
3-Methylphenol & 4-Methylphenol	10.0	ug/L	1.4	ug/L	20010124										
Methyl styrene	10.0	ug/L	2.7	ug/L	20010124										
Naphthalene	10.0	ug/L	1.4	ug/L	20010124										
1,4-Naphthoquinone	50.0	ug/L	1.0	ug/L	19960113										
1-Naphthylamine	10.0	ug/L	7.9	ug/L	19980113										
2-Naphthylamine	10.0	ug/L	7.6	ug/L	19980113										
2-Nitroaniline	50.0	ug/L	1.6	ug/L	20010124										
3-Nitroaniline	50.0	ug/L	6.7	ug/L	20010124										
4-Nitroaniline	50.0	ug/L	4.4	ug/L	20010124										
Nitrobenzene	10.0	ug/L	1.7	ug/L	20010124										
2-Nitrophenol	10.0	ug/L	1.7	ug/L	20010124										
4-Nitrophenol	50.0	ug/L	7.14	ug/L	20000302	150	ug/L	41	108	40	150	ug/L	41	108	40
4-Nitroquinoline-1-oxide	100	ug/L	15.77	ug/L	20000512										
N-Nitrosodi-n-butylamine	10.0	ug/L	1.5	ug/L	19960113										
N-Nitrosodiethylamine	10.0	ug/L	1.2	ug/L	19960113										
N-Nitrosodimethylamine	10.0	ug/L	1.7	ug/L	20010124										
N-Nftrosodiphenylamine	10.0	ug/L	5.3	ug/L	20010124										
N-Nitrosodi-n-propylamine	10.0	ug/L	1.8	ug/L	20010124	100	ug/L	48	88	33	100	ug/L	48	88	33
N-Niiosomethytethylamine	10.0	ug/L	1.0	ug/L	19980113										
N-Nitrosomorpholine	10.0	ug/L	1.5	ug/L	19989113										
N-Nitrosopiperidlne	10.0	ug/L	1.4	ug/L	19989113										
N-Nitrosopyrrolidine	10.0	ug/L	1.1	ug/L	19990113										
5-Nitro-o-toluidine	20.0	ug/L	3.2	ug/L	19986113										
Parathion	50.0	ug/L	1.3	ug/L	19980113										
Pentachlorobenzene	10.0	ug/L	1.4	ug/L	19980113										
Pentachloroethane	50.0	ug/L	1.4	ug/L	19980113										
Pentachloronitrobenzene	50.0	ug/L	1.0	ug/L	19960113										
Pentachlorophenol	50.0	ug/L	7.7	ug/L	20010124	150	ug/L	42	105	34	150	ug/L	42	105	34
Phenacetin	20.0	ug/L	1.0	ug/L	19969113										
Phenanthrene	10.0	ug/L	1.2	ug/L	20010124										
Phenol	10.0	ug/L	1.4	ug/L	29610124	150	ug/L	50	90	31	150	ug/L	50	90	31
p-Phenylene diamine			30.9	ug/L	20000512										
Phorate	50.0	ug/L	2.0	ug/L	19989113										
2Picoline	20.0	ug/L	1.4	ug/L	19960113										
Pronamide	20.0	ug/L	1.0	ug/L	19986113										
Pyrene	10.0	ug/L	1.7	ug/L	20010124	100	ug/L	54	104	31	100	ug/L	54	104	31
Pyridine	20.0	ug/L	12	ug/L	20010124										

Target Analyte List: DEN: 8270 'APO' List (includes TCL + AP9)

Matrix: WATER
Extraction: LIQ/LIQ, CONT (A/B/N) - Acid->Base
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL Denver

Target List 7066	Detection Limits					Check List 4340					Spike List 4340				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Safrole	50.0	ug/L	1.7	ug/L	19980113										
Sulfotepp	50	ug/L	1	ug/L	19980113										
1,2,4,5-Tetrachlorobenzene	10.0	ug/L	1.9	ug/L	19980113										
2,3,4,6-Tetrachlorophenol	50.0	ug/L	1.2	ug/L	19980113										
Thionazin	10	ug/L	1.15	ug/L	20000316										
2-Toluidine			4.6	ug/L	19980113										
1,2,4-Trichlorobenzene	10.0	ug/L	1.a	ug/L	20010124	100	ug/L	43	88	35	100	ug/L	43	88	35
2,4,5-Trichlorophenol	10.0	ug/L	1.1	ug/L	20010124										
2,4,6-Trichlorophenol	10.0	ug/L	1.1	ug/L	20010124										
O,O,O-Triethyl phosphordhioate	50.0	ug/L	1.6	ug/L	19980113										
1,3,5-Trinitrobenzene	50.0	ug/L	1.4	ug/L	19980113										
2-Fluorobiphenyl						100	ug/L	49	98	0	100	ug/L	40	08	0
2-Fluorophenol						150	ug/L	51	QQ	0	150	ug/L	51	99	0
2,4,6-Tribromophenol						150	ug/L	53	108	0	150	ug/L	53	108	0
Nitrobenzene-d5						100	ug/L	57	97	0	100	ug/L	57	97	0
Phenol-d5						150	ug/L	52	99	0	150	ug/L	52	99	0
Terphenyl-d14						100	ug/L	38	119	0	100	ug/L	38	119	0

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: WATER
Extraction: METALS, TOTAL - Waters
Method: Inductively Coupled Plasma (6010B)
QC Program: STANDARD TEST SET
Location: STL Denver

Analyte List		Detection Limits				Check List 4060					Spike List 4060				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aluminum	100	ug/L	22	ug/L	20010213	2.0	mg/L	89	109	20	2.0	mg/L	89	109	20.
Barium	10	ug/L	0.83	ug/L	20010117	2.0	mg/L	92	115	20	2.0	mg/L	92	115	20
Beryllium	5.0	ug/L	0.59	ug/L	20010117	0.050	mg/L	91	117	20	0.050	mg/L	91	117	20
Boron	100	ug/L	5.0	ug/L	20010117	1.0	mg/L	90	110	20	1.0	mg/L	90	110	20
Calcium	200	ug/L	29	ug/L	20010213	50	mg/L	88	108	20	50	mg/L	88	108	20
Chromium	10	ug/L	3.4	ug/L	20010117	0.20	mg/L	91	112	20	0.20	mg/L	91	i12	20
Copper	10	ug/L	2.9	ug/L	20010117	0.25	mg/L	93	113	20	0.25	mg/L	93	113	2 0
Iron	100	ug/L	5.1	ug/L	20010213	1.0	mg/L	92	114	20	1.0	mg/L	92	114	20
Lithium	10	ug/L	2.0	ug/L	20010117	1.0	mg/L	a7	108	20	1.0	mg/L	a7	108	20
Magnesium	200	ug/L	21	ug/L	20010117	50	mg/L	93	113	20	50	mg/L	93	113	20
Manganese	10	ug/L	0.66	ug/L	20010117	0.50	mg/L	89	114	20	0.50	mg/L	89	114	20
Nickel	40	ug/L	4.4	ug/L	20010213	0.50	mg/L	89	109	20	0.50	mg/L	89	lo9	20
Phosphorus	3ooo	ug/L	97	ug/L	20010117	10.0	mg/L	80	120	20	10.0	mg/L	Bo	120	20
Potassium	3000	ug/L	500	ug/L	20010117	50	mg/L	87	110	20	50	mg/L	87	110	20
Silica	500	ug/L	16	ug/L	20010117	21.4	mg/L	75	125	20	21.4	mg/L	75	125	20
Silicon	500	ug/L	16	ug/L	20010117	10	mg/L	76	125	20	10	mg/L	75	125	20
Sodium	5000	ug/L	2000	ug/L	20010117	50	mg/L	91	111	20	50	mg/L	91	111	20
strorltiurn	10	ug/L	0.38	ug/L	20010117	1.0	mg/L	a 9	109	20	1.0	mg/L	89	109	20
Titanium	10	ug/L	1.3	ug/L	20010117	1.0	mg/L	a2	112	10	1.0	mg/L	a2	112	lo
Vanadium	10	ug/L	3.8	ug/L	20010117	0.50	mg/L	88	115	20	0.50	mg/L	88	115	20
Zinc	20	ug/L	4.1	ug/L	20010117	0.60	mg/L	83	111	2 0	0.50	mg/L	83	111	20

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: WATER
Extraction: METALS, TOTAL -Waters
Method: inductively Coupled Plasma (601 0B Trace)
QC Program: STANDARD TEST SET
Location: STL Denver

Analyte List		Detection Limits			Check List 4075					Spike List 4075				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aluminum	100	ug/L	8.2	ug/L	20010125	2.0	mg/L	89	109	20	2.0	mg/L	69	109	20
Antimony	10	ug/L	3.1	ug/L	29010125	0.50	mg/L	86	106	20	0.50	mg/L	66	106	20
Arsenic	10	ug/L	4.3	ug/L	20010125	2.0	mg/L	90	110	20	2.0	mg/L	90	110	20
Barium	10	ug/L	0.64	ug/L	20010313	2.0	mg/L	92	115	20	2.0	mg/L	92	115	20
Beryllium	5.0	ug/L	0.22	ug/L	20010323	0.05	mg/L	91	117	20	0.05	mg/L	91	117	20
Cadmium	5.0	ug/L	0.29	ug/L	20010125	0.050	mg/L	91	111	20	0.050	mg/L	91	111	20
Chromium	10	ug/L	0.56	ug/L	20010221	0.20	mg/L	91	112	20	0.20	mg/L	91	112	20
Cobalt	10	ug/L	0.34	ug/L	20010221	0.5	mg/L	91	111	20	0.5	mg/L	91	111	20
Copper	10	ug/L	0.63	ug/L	20010125	0.25	mg/L	93	113	20	0.25	mg/L	93	113	20
Lead	3.0	ug/L	1.2	ug/L	20010125	0.50	mg/L	90	110	20	0.50	mg/L	90	110	20
Manganese	10	ug/L	0.38	ug/L	20010125	0.50	mg/L	89	114	io	0.50	mg/L	69	114	20
Molybdenum	20	ug/L	1.7	ug/L	20010125	1.0	mg/L	so	110	20	1.0	mg/L	90	110	20
Nickel	40	ug/L	0.96	ug/L	20010125	0.50	mg/L	a9	114	20	0.50	mg/L	89	114	20
Selenium	5.0	ug/L	4.5	ug/L	20010125	2.0	mg/L	91	111	20	2.0	mg/L	01	111	20
Silver	10	ug/L	0.82	ug/L	20010125	0.05	mg/L	94	114	20	0.05	mg/L	94	114	20
Sodium	1.0	mg/L	0.22	mg/L	20010711	50	mg/L	91	111	20	50	mg/L	91	111	20
Strontium	10	ug/L	0.18	ug/L	20010221	1.0	mg/L	89	109	20	1.0	mg/L	89	109	20
Thallium	10	ug/L	3.1	ug/L	20910125	2.0	mg/L	86	108	20	2.0	mg/L	68	108	20
Tin	100	ug/L	3.3	ug/L	20010125	2.0	mg/L	69	109	20	2.0	mg/L	69	109	20
Vanadium	10	ug/L	0.67	ug/L	20010221	0.50	mg/L	80	114	20	0.50	mg/L	60	114	20
Zinc	20	ug/L	6.6	ug/L	20010221	0.50	mg/L	77	113	20	0.50	mg/L	27	113	20

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: WATER
Extraction: METALS, TOTAL (Method exclusive) - Walers
Method: Mercury (7470A, Cold Vapor) - Liquid
QC Program: STANDARD TEST SET
Location: STL Denver

Analyte List		Detection Limits				Check List 4106					Splke List 4106				
Compound	RL	Units	MDL	Unitr	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Mercury	0.2	ug/L	0.030	ug/L	20010206	0.0050	mg/L	84	114	10	0.0054	mg/L	84	114	10

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: WATER
Extraction: REDUCTION
Method; Nitrate-Nitrite (353.2, Automated)
QC Program: STANDARD TEST SET
Location: sn Denver

Analyte List		Detection Limits				Check List 4215					Spike List 4215				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Nitrate/Nitrite	0.10	mg/L	0.021	mg/L	20000601	3.5	mg/L	90	110	10	3.5	mg/L	90	110	10

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: WATER
Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJ
Method: Sulfate (300.0, Ion Chromatography)
QC Program: STANDARDTESTSET
Location: STL Denver

Analyte List			Detection Limits			Check List 4217					Spike List 4217				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Sulfate	5.0	mg/L	0.10	mg/L	20001102	200	mg/L	90	110	10	200	mg/L	90	110	10

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: WATER
Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJ
Method: Chloride (300.0, Ion Chromatography)
QC Program: STANDARD TEST SET
Location: STL Denver

Analyte List Compound	RL	Detection Limits Units	MDL	Units	Run Date	Check List 4217 Amt	Units	LCL	UCL	RPD	Spike List 4217 Amt	Units	LCL	UCL	RPD
Chloride	3.0	mg/L	0.10	mg/L	20001102	200	mg/L	90	110	10	200	mg/L	90	110	10

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: WATER
Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJ
Method: Bromide (300.0, Ion Chromatography)
QC Program: STANDARD TEST SET
Location: STL Denver

Analyte List	Detection Limits					Check List 4217					Spike List 4217				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Bromide	0.20	mg/L	0.079	mg/L	20001102	20	mg/L	90	110	10	20	mg/L	00	110	10

STL Reference Data Summary

Target Analyte List: DEN: EPA VOC Appendix IX List

Matrix: SOLID
Extraction: PURGE AND TRAP - Lab Preserved Low Level - Solids
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL Denver

Target List 7061		Detection Limits				Check List 4350					Spike List 4350				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acetone	20	ug/kg	3.42	ug/kg	20000414										
Acetonitrile	100	ug/kg	16.80	ug/kg	20000414										
Acrolein	100	ug/kg	38.18	ug/kg	20000414										
Acrylonitrile	100	ug/kg	5.91	ug/kg	200004 14										
Benzene	5.0	ug/kg	0.50	ug/kg	20000414	50	ug/kg	79	121	25	50	ug/kg	79	121	25
Bromodichloromethane	5.0	ug/kg	0.50	ug/kg	20000414										
Bromoform	5.0	ug/kg	0.50	ug/kg	200004 14										
Bromomethane	10	ug/kg	0.50	ug/kg	20000414										
2-Butanone (MEK)	20	ug/kg	2.34	ug/kg	20000414										
tert-Butyl alcohol	200	ug/kg	12.55	ug/kg	20000414										
Carbon disulfide	5.0	ug/kg	0.52	ug/kg	20000414										
Carbon tetrachloride	5.0	ug/kg	0.54	ug/kg	20000414										
Chlorobenzene	5.0	ug/kg	1.01	ug/kg	20000414	50	ug/kg	76	116	25	50	ug/kg	76	116	25
Chloroprene	5.0	ug/kg	0.83	ug/kg	20000414										
Dibromochloromethane	5.0	ug/kg	0.50	ug/kg	20000414										
Chloroethane	10	ug/kg	0.50	ug/kg	20000414										
Chloroform	5.0	ug/kg	0.50	ug/kg	20000414										
Chlorornethane	10	ug/kg	0.91	ug/kg	20000414										
Allyl chloride	IO	ug/kg	0.54	ug/kg	20000414										
1,2-Dibromo-3-chloropropane (DBCP)	10	ug/kg	0.68	ug/kg	20000414										
1,2-Dibromoethane (EDB)	5.0	ug/kg	0.50	ug/kg	20000414										
Dibromomethane	5.0	ug/kg	*0.50*	ug/kg	20008414										
trans-1,4-Dichloro-2-butene	5.0	ug/kg	1.03	ughg	20000414										
Dichlorodifluoromethane	10	ug/kg	0.62	ug/kg	20000414										
1,1-Dichloroethane	5.0	ug/kg	0.65	ug/kg	20800414										
1,2-Dichloroethane	5.0	ug/kg	0.56	ug/kg	20000414										
cis-1,2-Dichloroethene	2.5	ug/kg	0.56	ug/kg	20000414										
trans-1,2-Dichloroethene	2.5	ug/kg	0.77	ug/kg	20000414										
1.1-Dichloroethene	5.0	ug/kg	0.71	ug/kg	20000414	50	ug/kg	78	118	25	50	ug/kg	78	118	25
1,2-Dichloroethene (total)	5.0	ug/kg	1.31	ug/kg	20000414										
1,2-Dichloropropane	5.0	ug/kg	0.50	ug/kg	20000414										
cis-1,3-Dichloropropene	5.0	ug/kg	0.72	ug/kg	20000414										
Irans-1,3-Dichloropropene	5.0	ug/kg	0.53	ug/kg	20000414										
1,4-Dioxane	500	ug/kg	42.82	ug/kg	20000414										
Ethylbenzene	5.0	ug/kg	1.14	ug/kg	20000414										
Ethyl methacrylate	5.0	ug/kg	0.60	ug/kg	20000414										
2-Hexanone	20	ug/kg	1.66	ug/kg	20000414										

Matrix: SOLID
Extraction: PURGE AND TRAP - Lab Preserved Low Level - Sdlds
Method: Volatile Organics, GC/MS (82606)
Target Analyte List: DEN: EPA VOC Appendix IX List
QC Program: STANDARD TEST SET
Localion: STL Denver

Target List 7061		Detection Limits				Check List 4350					Spike List 4350				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Iodomethane	5.0	ug/kg	0.50	ug/kg	20000414										
Isobutyl alcohol	200	ug/kg	11.66	ug/kg	20000414										
Methacrylonitrile	50	ug/kg	5.0	ug/kg	20006414										
Methylene chloride	5.0	ug/kg	0.50	ug/kg	20600414										
Methyl methacrytate	5.0	ug/kg	1.29	ug/kg	20000414										
4-Methyl-2-pentanone	20	ug/kg	1.20	ug/kg	266004 14										
Propionitrile	20	ug/kg	6.26	ug/kg	20000414										
Styrene	5.0	ug/kg	1.25	ug/kg	200604 14										
1,1,1,2-Tetrachloroethane	5.0	ug/kg	1.30	ug/kg	20060414										
1,1,2,2-Tetrachloroethane	5.0	ug/kg	0.50	ug/kg	26000414										
Tetrachloroethene	5.0	ug/kg	1.02	ug/kg	26006414										
Tduene	5.0	ug/kg	0.01	ug/kg	20006414	50	ug/kg	76	116	25	50	ug/kg	76	116	25
1,1,1-Trichloroethane	5.0	ug/kg	0.50	ug/kg	20000414										
1,1,2-Trichloroethane	5.0	ug/kg	0.90	ug/kg	26060414										
Trichloroethene	5.0	ug/kg	0.62	ug/kg	20000414	50	ug/kg	83	123	25	50	ug/kg	83	123	25
Trichlorofluoromethane	10	ug/kg	0.55	ug/kg	20060414										
1,2,3-Trichloropropane	5.0	ug/kg	1.13	ug/kg	20000414										
Vinyl acetate	10	ug/kg	4.38	ug/kg	20000414										
Vinyl chloride	10	ug/kg	0.76	ug/kg	20006414										
Xylenes (total)	5.0	ug/kg	3.08	ug/kg	20000414										
4-Bromofluorobenzene						50	ug/kg	71	132	0	50	ug/kg	71	132	0
1,2-Dichloroethane-d4						50	ug/kg	79	125	0	50	ug/kg	79	125	0
Toluene-d8						50	ug/kg	77	117	0	50	ug/kg	77	117	0
Dibrornofluoromethane						50	ug/kg	80	120	0	50	ug/kg	80	120	0

STL Reference Data Summary

Target Analyte List: DEN: 6270 "APB' List (includes TCL + AP9)

Matrix: SOLID
Extraction: SONICATION - Low Level
Method Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL Denver

Target List 7066		Detection Limits				Check List 4340					Spike List 4340				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acenaphthene	330	ug/kg	46	ug/kg	20010307	3330	ug/kg	49	93	40	3330	ug/kg	49	93	40
Acenaphthylene	330	ug/kg	34	ug/kg	20010367										
Acetophenone	330	ug/kg	33.0	ug/kg	20006314										
2-Acetylaminofluorene	3300	ug/kg	33.0	ug/kg	20606314										
4-Aminobiphenyl	1600	ug/kg	81.8	ug/kg	20000314										
Aniline	330	ug/kg	57	ug/kg	20610307										
Anthracene	330	ug/kg	78	ug/kg	20010307										
Aramite	660	ug/kg	66.0	ug/kg	20660314										
Azobenzene	330	ug/kg	65	ug/kg	20010307										
Benzidine	3300	ug/kg	680	ug/kg	20010307										
Benzo(a)anthracene	330	ug/kg	39	ug/kg	20010307										
Benzo(b)fluoranthene	330	ug/kg	100	ug/kg	20010367										
Benzo(k)fluoranthene	330	ug/kg	93	ug/kg	20610307										
Benzoic acid	1600	ug/kg	570	ug/kg	20010307										
Benzo(ghi)perylene	330	ug/kg	70	ug/kg	20010307										
Benzo(a)pyrene	330	ug/kg	94	ug/kg	26010307										
Benzyl alcohol	330	ug/kg	77	ug/kg	20010307										
bis(2-Chloroethoxy)methane	330	ug/kg	74	ug/kg	20010307										
bis(2-Chloroethyl) ether	330	ug/kg	49	ug/kg	20010307										
bis(2-Chloroisopropyl) ether	330	ug/kg	69	ug/kg	26010307										
bis(2-Ethylhexyl) phthalale	330	ug/kg	69	ug/kg	26010307										
4-Bromophenyl phenyl ether	330	ug/kg	71	ug/kg	26010307										
Butyl benzyl phthalate	330	ug/kg	34	ug/kg	20010367										
Carbazole	330	ug/kg	58	ug/kg	20610307										
4-Chloroaniline	330	ug/kg	47	ug/kg	20010307										
Chlorobenzilate	330	ug/kg	36.5	ug/kg	26090314										
4-Chloro-3-methylphenol	330	ug/kg	95	ug/kg	26010307	5000	ug/kg	52	93	40	5000	ug/kg	52	93	40
1-Chloronaphthalene	2500	ug/kg	33.0	ug/kg	20000314										
2-Chloronaphthalene	330	ug/kg	36	ug/kg	26010367										
2-Chlorophenol	330	ug/kg	73	ug/kg	29010307	5000	ug/kg	51	91	36	5000	ug/kg	51	91	36
4-Chlorophenyl phenyl ether	330	ug/kg	71	ug/kg	20010307										
Chtysene	330	ug/kg	53.2	ug/kg	20910367										
Diallate	660	ug/kg	66.0	ug/kg	20000314										
Dibenz(a,j)acridine	660	ug/kg	33.0	ug/kg	20000314										
Dibenz(a,h)anthracene	330	ug/kg	47	ug/kg	20010307										
Dibenzofuran	330	ug/kg	82	ug/kg	20010307										
Di-n-butyl phthalate	330	ug/kg	76	ug/kg	20010307										

Target Analyte List: DEN: 8270 "AP9" List (includes TCL + AP9)

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL Denver

Target Lfst 7066	Detection Limits					Check List 4340					Spike List 4340				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
1,2-Dichlorobenzene	330	ug/kg	64	ug/kg	20010307										
1,3-Dichlorobenzene	330	ug/kg	71	ug/kg	20010307										
1,4-Dichlorobenzene	330	ug/kg	55	ug/kg	20010307	3330	ug/kg	46	66	40	3330	ug/kg	46	66	40
3,3'-Dichlorobenzidine	1600	ug/kg	70	ug/kg	20010307										
2,4-Dichlorophenol	330	ug/kg	66	ug/kg	20010307										
2,6-Dichlorophenol	330	ug/kg	33.0	ug/kg	20000314										
Diethyl phthalate	660	ug/kg	53	ug/kg	20010307										
Dimethoate	660	ug/kg	33.5	ug/kg	20000314										
p-Dimelhylaminoazobenzene			42.3	ug/kg	20000314										
7,12-Dimethylbenz(a)anthracene	660	ug/kg	37.0	ug/kg	20000314										
3,3'-Dimethylbenzidine	660	ug/kg	57.0	ug/kg	20000314										
alpha,alpha-Dimethylphenethylamine	1600	ug/kg	174	ug/kg	19991130										
2,4-Dimethylphenol	330	ug/kg	92	ug/kg	20010307										
Dimethyl phthalate	330	ug/kg	85	ug/kg	20010307										
1,3-Dinitrobenzene	330	ug/kg	42.4	ug/kg	20000314										
1,4-Dinitrobenzene	330	ug/kg	33.0	ug/kg	20000314										
4,6-Dinitro-2-methylphenol	1600	ug/kg	420	ug/kg	20010307										
2,4-Dinitrophenol	1600	ug/kg	500	ug/kg	20010307										
2,4-Dinitrotoluene	330	ug/kg	96	ug/kg	20010307	5000	ug/kg	53	105	40	5000	ug/kg	53	105	40
2,6-Dinitrotoluene	330	ug/kg	100	ug/kg	20010307										
2-sec-Butyl-4,6-dinitrophenol	660	ug/kg	33.0	ug/kg	20000314										
Di-n-octyl phthalate	330	ug/kg	36	ug/kg	20010307										
Disulfoton	1600	ug/kg	33.0	ug/kg	20000314										
Ethyl methanesulfonate	330	ug/kg	44.5	ug/kg	20000314										
Fluoranthene	330	ug/kg	84	ug/kg	20010307										
Fluorene	330	ug/kg	76	ug/kg	20010307										
Hexachlorobenzene	330	ug/kg	76	ug/kg	26010307										
Hexachlorobutadiene	330	ug/kg	100	ug/kg	20010307										
Hexachlorocyclopentadiene	1600	ug/kg	33.0	ug/kg	20000228										
Hexachtoroethane	330	ug/kg	50	ug/kg	20010367										
Hexachloropropene	3300	ug/kg	40.0	ug/kg	20006314										
tndene	330	ug/kg	48	ug/kg	20010307										
Indeno(1,2,3-cd)pyrene	330	ug/kg	48	ug/kg	20010307										
Isodrin	330	ug/kg	33.0	ug/kg	20600314										
tsophorone	330	ug/kg	68	ug/kg	20010307										
Isosafrole	660	ug/kg	68.0	ug/kg	20000314										
Methapyrilene	1606	ug/kg	56.7	ug/kg	20000314										
3-Methylcholanthrene	660	ug/kg	33.0	ug/kg	20000314										
Methyl methanesulfonate	330	ug/kg	36.3	ug/kg	20000314										
2-Methylnaphthalene	330	ug/kg	59	ug/kg	20010307										

Target Analyte List: DEN: 8270 "AP9" List (includes TCL + AP9)

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL Denver

Target List 7066		Detection Limits				Check List 4340					Spike List 4340				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
1-Methylnaphthalene	330	ug/kg	36	ug/kg	20010307										
Methyl paralhlon	1600	ug/kg	33.6	ug/kg	20000314										
2-Methylphenol	330	ug/kg	77	ug/kg	20010307										
3-Methylphenol & 4-Methylphenol	330	ug/kg	74	ug/kg	20010307										
Methyl styrene	330	ug/kg	71	ug/kg	20010307										
Naphthalene	330	ug/kg	70	ug/kg	20010307										
1,4-Naphthoquinone	1600	ug/kg	33.0	ug/kg	20000314										
1-Naphthylamine	330	ug/kg	83.9	ug/kg	20000314										
2-Naphthylamine	330	ug/kg	77.9	ug/kg	20000314										
2-Nitroaniline	1600	ug/kg	80	ug/kg	20010307										
3-Nitroaniline	1600	ug/kg	a5	ug/kg	20610307										
4-Nitroaniline	1600	ug/kg	64	ug/kg	20610307										
Nitrobenzene	330	ug/kg	85	ug/kg	20010307										
2-Nitrophenol	330	ug/kg	120	ug/kg	20010307										
4-Nitrophenol	1600	ug/kg	95	ug/kg	20010307	5000	ug/kg	29	115	40	5000	ug/kg	29	115	40
4-Nitroquinoline-1-oxide			42.7	ug/kg	20000314										
N-Nitrosodi-n-butylamine	330	ug/kg	33.0	ug/kg	20000314										
N-Nitrosodiethylamine	330	ug/kg	37.0	ug/kg	20000314										
N-Nitrosodimethylamine	330	ug/kg	59	ug/kg	20010307										
N-Nitrosodiphenylamine	330	ug/kg	72	ug/kg	20010307										
N-Nitrosodi-n-propylamine	330	ug/kg	88	ug/kg	20010307	3330	ughg	46	86	40	3330	ug/kg	46	86	40
N-Nitrosomethylethylamine	330	ug/kg	45.9	ug/kg	20006314										
N-Nitrosomorpholine	330	ug/kg	52.7	ug/kg	20000314										
N-Nitrosopiperidine	330	ug/kg	33.0	ug/kg	20000314										
N-Nitrosopyrrolidine	330	ug/kg	36.6	ug/kg	20000314										
5-Nitro-o-toluidine	660	ug/kg	58.8	ug/kg	20000314										
Parathion	1600	ug/kg	70.0	ug/kg	20000314										
Pentachlorobenzene	330	ug/kg	33.0	ug/kg	20000314										
Pentachloroethane	1600	ug/kg	33.0	ug/kg	20000314										
Pentachloronitrobenzene	1600	ug/kg	33.0	ug/kg	20000314										
Pentachlorophenol	1600	ug/kg	370	ug/kg	20010307	5000	ug/kg	27	97	40	5000	ug/kg	27	97	40
Phenacetin	660	ug/kg	48.8	ug/kg	20000314										
Phenanthrene	330	ug/kg	37	ug/kg	20010307										
Phenol	330	ug/kg	71	ug/kg	20010307	5000	ug/kg	50	90	37	5000	ug/kg	50	90	37
p-Phenylene diamine			839	ug/kg	19991021										
Phorate	1600	ug/kg	33.0	ug/kg	20000314										
2-Picoline	660	ug/kg	33.0	ug/kg	20000314										
Pronamide	660	ug/kg	33.0	ug/kg	20000314										
Pyrene	330	ug/kg	40	ug/kg	20610307	3330	ug/kg	48	97	40	3330	ug/kg	48	97	40
Pyridlne	660	ug/kg	400	ug/kg	20010307										

Target Analyte List: DEN: 8270 "AP9" List (includes TCL + AP9)

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals end Acids (827oC)
QC Program: STANDARDTESTSET
Location: STL Denver

Target List 7066	Detection Limits					Check List 4340					Spike List 4340				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Sefrole	1600	ug/kg	36.7	ug/kg	20000314										
Sulfotepp	1000	ug/kg	33.0	ug/kg	20000314										
1,2,4,5-Tetrachlorobenzene	330	ug/kg	33.0	ug/kg	zoo00314										
2,3,4,6-Tetrachlorophenol	1600	ug/kg	47.9	ug/kg	20000314										
Thionazin	1600	ug/kg	48.2	ug/kg	20000314										
2-Toluidine	660	ug/kg	98.3	ug/kg	20000314										
1,2,4-Trichlorobenzene	330	ug/kg	64	ug/kg	20010307	3330	ug/kg	49	90	40	3330	ug/kg	49	90	40
2,4,5-Trichlorophenol	330	ug/kg	75	ug/kg	20010307										
2,4,6-Trichlorophenol	330	ug/kg	50	ug/kg	20010307										
O,O,O-Triethylphosphorolhloate	1600	ug/kg	35.1	ug/kg	20000314										
1,3,5-Trinitrobenzene	1600	ug/kg	48.2	ug/kg	20000314										
2-Fluorobiphenyl						3330	ug/kg	39	91	0	3330	ug/kg	39	91	0
2-Fluorophenol						3330	ug/kg	34	97	0	3330	ug/kg	34	97	0
2,4,6-Tribromophenol						5000	ug/kg	29	95	0	5000	ug/kg	29	95	0
Nitrobenzene-d5						3330	ug/kg	33	97	0	3330	ug/kg	33	97	0
Phenol-d5						5000	ug/kg	39	90	0	5000	ug/kg	39	90	0
Terphenyl-d14						3330	ug/kg	30	102	0	3330	ug/kg	30	102	0

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: METALS, TOTAL - Soiis
Method: Inductively Coupled Plasma (601 OB Trace)
QC Program: STANDARD TEST SET
Location: STL Denver

Analyte List		Detection Limits			Check List 4075					Spike List 4075				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Antimony	1.0	mg/kg	0.51	mg/kg	20010117	50	mg/kg	a5	105	20	50	mg/kg	85	105	20
Arsenic	1.0	mg/kg	0.29	mg/kg	20010117	200	mg/kg	a7	107	20	200	mg/kg	a7	107	20
Barium	1.0	mg/kg	0.088	mg/kg	20010418	200	mg/kg	86	114	20	200	mg/kg	86	114	20
Beryllium	0.5	mg/kg	0.029	mg/kg	20010418	5	mg/kg	90	110	20	5	mg/kg	90	110	20
Cadmium	0.5	mg/kg	0.033	mg/kg	20010212	5	mg/kg	89	109	20	5	mg/kg	a9	109	20
Calcium	20	mg/kg	2.9	mg/kg	20010212	5000	mg/kg	a9	109	20	5000	mg/kg	a9	109	20
Chromium	1.0	mg/kg	0.035	mg/kg	20010117	20	mg/kg	88	110	20	20	mg/kg	88	110	20
Cobalt	1	mg/kg	0.067	mg/kg	20010212	50	mg/kg	06	106	20	50	mg/kg	86	106	20
Copper	2.0	mg/kg	0.091	mg/kg	20010117	25	mg/kg	90	110	20	25	mg/kg	90	110	20
Lead	0.8	mg/kg	0.21	mg/kg	20010117	50	mg/kg	88	108	20	50	mg/kg	88	foa	20
Magnesium	20	mg/kg	2.0	mg/kg	20010418	5000	mg/kg	91	113	20	5000	mg/kg	91	113	20
Manganese	1.0	mg/kg	0.027	mg/kg	20010330	50	mg/kg	90	110	20	50	mg/kg	90	110	20
Molybdenum	2	mg/kg	0.16	mg/kg	20010117	100	mg/kg	87	107	20	100	mg/kg	a7	107	20
Nickel	4.0	mg/kg	0.11	mg/kg	20010212	50	mg/kg	88	108	20	50	mg/kg	88	108	20
Selenlurn	1.3	mg/kg	0.39	mg/kg	20010117	200	mg/kg	86	107	20	200	mg/kg	86	107	20
Silver	1.0	mg/kg	0.071	mg/kg	20010117	5	mg/kg	88	108	20	5	mg/kg	88	108	20
Strontium	1.0	mg/kg	0.012	mg/kg	20010212	100	mg/kg	89	109	20	100	mg/kg	a9	109	20
Thallium	1.2	mg/kg	0.36	mg/kg	20010117	200	mg/kg	85	105	20	200	mg/kg	a5	105	20
Tin	10	mg/kg	0.17	mg/kg	20010212	200	mg/kg	84	108	20	200	mg/kg	84	108	20
Vanadium	1.0	mg/kg	0.060	mg/kg	20010117	50	mg/kg	88	110	20	50	mg/kg	88	110	20
Zinc	2.0	mg/kg	0.14	mg/kg	20010330	50	mg/kg	86	107	20	50	mg/kg	a6	107	20

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: METALS, TOTAL'- Soils
Method: Inductively Coupled Plasma (6010B)
QC Program: STANDARD TEST SET
Location: STL Denver

Analyte List		Detection Limits				Check List 4060					Spike List 4060				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Aluminum	10	mg/kg	1.9	mg/kg	20010117	200	mg/kg	07	107	20	200	mg/kg	87	107	20
Barium	1.o	mg/kg	0.050	mg/kg	20010213	200	mg/kg	86	114	20	200	mg/kg	a6	114	20
Beryllium	0.5	mg/kg	0.042	mg/kg	20010213	5.0	mg/kg	90	110	20	5.0	mg/kg	90	110	20
Boron	10	mg/kg	0.97	mg/kg	20010117	100	mg/kg	85	125	2 0	100	mg/kg	85	125	20
Calcium	20	mg/kg	4.2	mg/kg	20010213	5000	mg/kg	89	109	20	5000	mg/kg	a9	109	20
Chromium	1.0	mg/kg	0.23	mg/kg	20010213	20	mg/kg	88	110	20	20	mg/kg	88	110	20
Copper	2.0	mg/kg	0.35	mg/kg	20010117	25	mg/kg	90	110	20	25	mg/kg	90	110	20
Iron	10	mg/kg	0.58	mg/kg	20010117	100	mg/kg	90	116	20	100	mg/kg	90	116	20
Lithium	5.0	mg/kg	0.20	mg/kg	20010117	100	mg/kg	80	120	2 0	100	mg/kg	80	120	20
Magnesium	20	mg/kg	1.8	mg/kg	20010117	5000	mg/kg	91	113	20	5000	mg/kg	91	113	20
Manganese	1.0	mg/kg	0.065	mg/kg	20010117	50	mg/kg	Qo	110	20	50.	mg/kg	90	110	20
Nickel	4.0	mg/kg	0.39	mg/kg	20010117	50	mg/kg	88	108	2 0	50	mg/kg	88	108	20
Phosphorus	300	mg/kg	5.0	mg/kg	20010117			75	125	2 0			75	125	20
Potassium	300	mg/kg	65	mg/kg	26010117	5000	mg/kg	86	112	20	5000	mg/kg	86	112	20
Sodium	500	mg/kg	130	mg/kg	20010117	5000	mg/kg	81	116	20	5ooo	mg/kg	81	115	20
Strontium	1.0	mg/kg	0.028	mg/kg	20010117	100	mg/kg	80	120	20	100	mg/kg	80	120	20
Titanium	1.0	mg/kg	0.064	mg/kg	20610117	100	mg/kg	a0	120	20	100	mg/kg	80	120	20
Vanadium	1.0	mg/kg	0.23	mg/kg	20010117	50	mg/kg	88	110	20	50	mg/kg	88	110	20
Zinc	2.6	mg/kg	0.41	mg/kg	20010213	50	mg/kg	86	107	20	50	mg/kg	86	107	20

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: METALS, TOTAL - Soils
Method: Inductively Coupled Plasma Mass Spectrometry(6020)
QC Program: STANDARD TEST SET
Location: STL Denver

Analyte List		Detection Limits				Check List 4063					Spike List 4063				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Antimony	200	ug/kg	22	ug/kg	20010117	20	mg/kg	92	126	20	20	mg/kg	92	126	20
Arsenic	500	ug/kg	51	ug/kg	20010117	20	mg/kg	91	113	20	20	mg/kg	91	113	20
Barium	100	ug/kg	14	ug/kg	20910663	20	mg/kg	80	120	20	20	mg/kg	80	120	20
Beryllium	100	ug/kg	26	ug/kg	20010603	20	mg/kg	84	117	20	20	mg/kg	84	117	20
Cadmium	100	ug/kg	6.3	ug/kg	20010319	20	mg/kg	90	111	20	20	mghg	90	111	20
Chromium	200	ug/kg	60	ug/kg	20010117	20	mg/kg	85	115	20	20	mg/kg	85	115	20
Cobalt	100	ug/kg	2.5	ug/kg	20010503	20	mg/kg	a6	111	20	20	mg/kg	86	111	20
Copper	200	ug/kg	20	ug/kg	20610319	20	mg/kg	91	113	20	20	mg/kg	91	113	20
Lead	100	ug/kg	21	ug/kg	20010117	20	mg/kg	89	116	20	20	mg/kg	89	116	20
Manganese	100	ug/kg	29	ug/kg	20010117	20	mg/kg	80	120	20	20	mg/kg	80	120	20
Molybdenum	200	ug/kg	25	ug/kg	20010117	20	mg/kg	80	120	20	20	mg/kg	80	120	20
Nickel	100	ug/kg	11	ug/kg	20010319	20	mg/kg	90	110	20	20	mg/kg	90	110	20
Selenium	500	ug/kg	62	ug/kg	20010117	20	mg/kg	83	117	20	20	mg/kg	63	117	20
Silver	100	ug/kg	5.0	ug/kg	20010503	20	mg/kg	81	114	20	20	mg/kg	81	114	20
Thallium	100	ug/kg	2.0	ug/kg	20910503	20	mg/kg	83	115	20	20	mg/kg	63	115	20
Tin	1000	ug/kg	74	ug/kg	20910117	20	mg/kg	80	120	20	20	mg/kg	80	120	20
Uranium	100	ug/kg	2.0	ug/kg	20010603	20	mg/kg	80	120	20	20	mg/kg	80	120	20
Vanadium	500	ug/kg	12	ug/kg	20010319	20	mg/kg	80	120	20	20	mghg	80	120	20
Zinc	1000	ug/kg	570	ug/kg	20010117	20	mg/kg	86	116	20	20	mg/kg	66	116	20

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: METALS, TOTAL (Method Exclusive) - Solids
Method: Mercury (7471A, Cold Vapor) - Solids
QC Program: STANDARD TEST SET
Location: STL Denver

Analyte List		Detection Limits			Check List 4106					Spike List 4106				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Mercury	0.033	mg/kg	0.0026	mg/kg	20010215	0.4167	mg/kg	62	113	20	0.416;	mg/kg	82	113	20

STL Reference Data Summary

Target Analyte List: DEN: 8260 EPA TCLP list

Matrix: SOLID
Extraction: TCLP(1311-ZHE/filter) -> PURGE-AND-TRAP (Low Level)
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL Denver

Target List 7031	Detection Limits					Check List 4353					Spike List 4353				
Compound	RL	Units	MDL	Units	Run Date	Amt	Untts	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Benzene	0.010	mg/L	0.002 1	mg/L	20000313	0.50	mg/L	79	119	20	0.50	mg/L	79	119	20
2-Butanone	0.050	mg/L	0.0093	mg/L	20000313	0.50	mg/L	26	181	33	0.50	mg/L	26	161	33
Carbon tetrachloride	0.010	mg/L	0.0019	mg/L	20000314	0.50	mg/L	84	124	20	0.50	mg/L	84	124	20
Chlorobenzene	0.010	mg/L	0.0030	mg/L	20300313	0.50	mg/L	76	116	20	0.50	mg/L	76	116	20
Chloroform	0.010	mg/L	0.0023	mg/L	20000313	0.50	mg/L	80	120	20	0.50	mg/L	80	120	20
1,2-Dichloroethane	0.010	mg/L	0.0028	mg/L	20000313	0.50	mg/L	77	122	20	0.50	mg/L	77	122	20
1,1-Dichloroethene	0.010	mg/L	0.0020	mg/L	20000313	0.50	mg/L	79	119	20	0.50	mg/L	79	119	20
Telrachloroethene	0.010	mg/L	0.0036	mg/L	20000314	0.50	mg/L	83	123	20	0.50	mg/L	83	123	20
Trichloroethene	0.010	mg/L	0.0022	mg/L	20000314	0.50	mg/L	81	121	20	0.50	mg/L	81	121	20
Vinyl chloride	0.010	mg/L	0.0021	mg/L	20000314	0.50	mg/L	68	124	20	0.50	mg/L	68	124	20
4-Bromofluorobenzene						0.50	mg/L	79	119	0	0.50	mg/L	79	119	0
1,2-Dichloroethane-d4						0.50	mg/L	76	122	0	0.50	mg/L	76	122	0
Toluene-d8						0.50	mg/L	79	119	0	0.50	mg/L	79	119	0
Dibromofluoromethane						0.50	mg/L	80	120	0	0.50	mg/L	80	120	0

STL Reference Data Summary

Target Analyte List: DEN: 8270 TCLP list

Matrix: SOLID
Extraction: TCLP(1311) -> LIQ/LIQ, CONT - Acid->Base
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL Denver

Target List 7032		Detection Limits			Check List 4017					Spike List 4017				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
1,4-Dichlorobenzene	0.1	mg/L	0.01045	mg/L	20000302	0.250	mg/L	36	101	30	0.250	mg/L	36	101	30
2,4-Dinitrotoluene	0.1	mg/L	0.00720	mg/L	20000302	0.250	mg/L	50	102	45	0.250	mg/L	50	102	45
Hexachlorobenzene	0.1	mg/L	0.01365	mg/L	20000302	0.250	mg/L	52	114	37	0.250	mg/L	52	114	37
Hexachlorobutadiene	0.1	mg/L	0.01125	mg/L	20000302	0.260	mg/L	32	109	27	0.250	mg/L	32	109	27
Hexachloroethane	0.1	mg/L	0.0066	mg/L	20000302	0.250	mg/L	32	104	27	0.250	mg/L	32	104	27
2-Methylphenol	0.1	mg/L	0.0159	mg/L	20000302	0.25	mg/L	43	103	50	0.25	mg/L	43	103	50
3-Methylphenol & 4-Methylphenol	0.1	mg/L	0.01635	mg/L	20000302	0.50	mg/L	41	101	45	0.50	mg/L	41	101	45
Nitrobenzene	0.1	mg/L	0.01225	mg/L	20000302	0.250	mg/L	50	109	39	0.250	mg/L	50	109	39
Pentachlorophenol	0.25	mg/L	0.025	mg/L	20000314	0.50	mg/L	20	119	50	0.50	mg/L	20	119	50
Pyridine	0.1	mg/L	0.0302	mg/L	20000313	0.250	mg/L	20	96	39	0.250	mg/L	20	96	39
2,4,5-Trichlorophenol	0.1	mg/L	0.01225	mg/L	20000302	0.250	mg/L	43	99	45	0.250	mg/L	43	99	45
2,4,6-Trichlorophenol	0.1	mg/L	0.01135	mg/L	20000302	0.260	mg/L	36	97	36	0.250	mg/L	36	97	36
2-Fluorobiphenyl						0.50	mg/L	33	99	0	0.50	mg/L	33	99	0
2-Fluorophenol						0.75	mg/L	38	91	0	0.75	mg/L	38	91	0
2,4,6-Tribromophenol						0.75	mg/L	40	110	0	0.75	mg/L	40	110	0
Nitrobenzene-d5						0.50	mg/L	41	107	0	0.50	mg/L	41	107	0
Phenol-d5						0.75	mg/L	37	94	0	0.75	mg/L	37	94	0
Terphenyl-d14						0.50	mg/L	47	107	0	0.50	mg/L	47	107	0

STL Reference Data Summary

Target Analyte List: DEN: 8080/8081A TCLP

Matrix: SOLID
Extraction: TCLP(1311) -> LIQ/LIQ, SEP FUNNEL - Nominal
Method: Pesticides (8081 A)
QC Program: STANDARD TEST SET
Location: STL Denver

Target List 7034	Detection Limits					Check List 4155				Spike List 4155				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
gamma-BHC (Lhdane)	0.00050	mg/L	0.00005	mg/L	20010111	0.0050	mg/L	68	142	30	0.005(	mg/L	68	142	30
Chlordane (technical)	0.0050	mg/L	0.00088	mg/L	20001106										
Endrin	0.0005	mg/L	0.00006	mg/L	20010111	0.0050	mg/L	66	143	30	0.005(	mg/L	66	143	30
Heptachlor	0.00050	mg/L	0.00008	mg/L	20010111	0.0050	mg/L	59	143	30	0.005(	mg/L	59	143	30
Heptachlor epoxide	0.00050	mg/L	0.09005	mg/L	20010111	0.0050	mg/L	37	142	30	0.005(	mg/L	37	142	30
Methoxychlor	0.901	mg/L	0.00009	mg/L	29010111	0.0050	mg/L	30	150	30	0.005(	mg/L	30	150	30
Toxaphene	0.02	mg/L	0.0051	mg/L	20010601										
Decachlorobiphenyl						0.0020	mg/L	50	151	0	0.002(	mg/L	50	151	0
Tetrachloro-m-xylene						0.0020	mg/L	64	131	0	0.002(	mg/L	64	131	0

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STL Reference Data Summary

Target Analyte List: DEN: 8150 TCLP List

Matrix: SOLID
Extraction: TCLP(1311) -> LIQ/LIQ, SEP FUNNEL - Acid -> DERIVATIZ
Method: Herbicides (8151A)
QC Program: STANDARD TEST SET
Location: STL Denver

Target List 7039		Detection Limits				Check List 4330					Spike List 4330				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
2,4-D	0.04	mg/L	0.003	mg/L	19990606	160	ug/kg	20	114	40	160	ug/kg	20	114	40
2,4,5-TP (Silvex)	0.01	mg/L	0.0001	mg/L	19990506	40	ug/kg	28	126	40	40	ug/kg	28	126	40
DCAA						0.100	ug/kg	20	133	0	0.100	ug/kg	20	133	0

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: TCLP(1311) -> METALS, TOTAL
Method: Inductively Coupled Plasma (6010B Trace)
QC Program: STANDARD TEST SET
Location: STL Denver

Analyte List			Detection Limitr			Check List 4077					Spike List 4077				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Antimony	0.10	mg/L	3.1	ug/L	20010125	0.50	mg/L	80	120	20	0.50	mg/L	80	120	20
Arsenic	0.5	mg/L	4.3	ug/L	20010125	7	mg/L	86	116	15	7	mg/L	66	116	15
Barium	IO	mg/L	0.64	ug/L	20010313	52	mg/L	85	114	15	52	mg/L	85	114	15
Beryllium	0.050	mg/L	0.22	ug/L	20010323	0.05	mg/L	80	120	20	0.05	mg/L	80	120	20
Cadmium	0.1	mg/L	0.29	ug/L	20010125	1.05	mg/L	88	114	15	1.05	mg/L	88	114	15
Chromium	0.5	mg/L	0.56	ug/L	20010221	5.2	mg/L	87	111	15	5.2	mg/L	87	111	15
Cobalt	0.10	mg/L	0.34	ug/L	20010221	0.5	mg/L	80	120	20	0.5	mg/L	80	120	20
Copper	0.10	mg/L	0.83	ug/L	20010125	0.25	mg/L	85	111	IO	0.25	mg/L	85	111	10
Lead	0.5	mg/L	1.2	ug/L	20010125	5.5	mg/L	85	116	15	5.5	mg/L	85	116	15
Manganese	0.10	mg/L	0.38	ug/L	20010126	0.5	mg/L	89	114	20	0.5	mg/L	89	114	20
Molybdenum	0.20	mg/L	1.7	ug/L	20010125	1.0	mg/L	80	120	20	1.0	mg/L	80	120	20
Nickel	0.40	mg/L	0.96	ug/L	20010125	0.5	mg/L	80	120	20	0.5	mg/L	80	120	20
Selenium	0.25	mg/L	4.5	ug/L	20010125	3.0	mg/L	87	118	15	3.0	mg/L	87	118	15
Silver	0.50	mg/L	0.62	ug/L	20010125	1.05	mg/L	86	114	15	1.05	mg/L	86	114	15
Thallium	0.10	mg/L	3.1	ug/L	20010125	2.0	mg/L	80	120	20	2.0	mg/L	80	120	20
Vanadium	0.10	mg/L	0.67	ug/L	20010221	0.5	mg/L	80	120	20	0.5	mg/L	80	120	20
Zinc	0.20	mg/L	6.6	ug/L	20010221	0.5	mg/L	75	139	20	0.5	mg/L	75	139	20

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: TCLP(I31 I) -> METALS, TOTAL (Method exclusive)
Math& Mercury (7470A, Cold Vapor) - Liquid
QC Program: STANDARD TEST SET
Location: STL Denver

Analyte List		Detection Limits				Check List 4108					Spike List 4108				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Mercury	0.002	mg/L	0.030	ug/L	20010206	0.005	mg/L	80	111	10	0.005	mg/L	60	111	10

STANDARD OPERATING PROCEDURE

TITLE: LC/MS ANALYSIS OF NITROAROMATIC AND NITRAMINE EXPLOSIVE COMPOUNDS BY APCI/LC/MS

(SUPERSEDES: Revision 1)

Prepared by: Larry Penfold

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Laboratory Director, Timothy O'Shields

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3.1. The quality control terminology used in this procedure follow SW-846 conventions, which are defined in the glossary of STL Denver's Laboratory Quality Manual (LQM).

4. INTERFERENCES

4.1. LC/MS is generally highly selective for the analytes of interest. LC/MS applies three means of discrimination:

a) chromatographic separation on the LC column,
b) generation of negative ions (typical organic molecular ions are most stable as positive ions, whereas nitroaromatics are stable as a negative ion), and
c) mass, selection.

The procedure allows close monitoring of retention time shifts because it employs two internal standards and one surrogate compound, as compared to a single surrogate typically used with the conventional HPLC method (8330). Although LC/MS is more selective than traditional HPLC, it is theoretically possible that compounds could have similar retention times on the LC column and generate negative ions with an identical mass as one of the target compounds. Use of the second quadrupole (LC/MS/MS) is an option that allows further fragmentation of the parent ion and unambiguous compound identification.

4.2. Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw LC/MS/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples and then take corrective action to eliminate the problem.

4.3. The use of high purity reagents, solvents; and gases helps to minimize interference problems.

4.4. Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

5. SAFETY PRECAUTIONS

5.1. Procedures shah be carried out in a manner that protects the health and safety of all STL personnel associates.

5.2. Acetonitrile and methanol are flammable solvents. Analysts should be aware of the safety precautions for flammable solvents outlined in the Chemical Hygiene Plan (CHP) and in the Materials Safety Data Sheets (MSDSs).

5.3. The following requirements must be met:

5.3.1. Eye protection that satisfies ANSI 287.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposabfe gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3.2. Soil samples with concentrations higher than 2 % should not be ground in the mortar and pestle. Visual observations of a soil sample is also important when taken from a site expected to contain explosives. Lumps of material that have a chemical appearance should be suspect and not ground.

5.3.3. Chemicals **known** to be **flammable are:**

Methanol, and acetonitrile.

5.3.4. The following materials **are known** to be **corrosive:**

Sulfuric acid.

5.3.5. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory.

5.3.6. Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.

5.4. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported immediately to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Liquid Chromatograph/Mass Spectrometer System

An analytical system complete with a ternary gradient programmable high-pressure liquid chromatograph and a column heater capable of maintaining a temperature of 15° +/- 1 °C. The mass spectrometer system must have an APCI interface capable of operation at 0.25 mLs per minute flow and be capable of negative ion analysis.

6.2. Chromatography Column

A 250 X 3.2 mm C_{18} reversed phase column. Alternate columns are acceptable if they provide acceptable performance.

6.3. Data System

A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any LC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits.

6.4. Autosampler

An autosampler that is capable of injecting 50µL with a precision of 5% RSD and that can accommodate at least 96 samples.

6.5. Balance, capable of measuring ± 0.01 g.

6.6. pH paper, wide range.

6.7. Graduated cylinders, 100 mL, 500 mL, and 1,000 mL

6.8. Media bottles, 500 mL.

6.9. Sonicator bath, with at least a 200 watt rating.

6.10. Various vials:

. Glass, 40 mL

- Amber with Teflon-lined screw caps, 12.5 mL
- Amber with Teflon-lined screw caps, 4 mL
- Amber crimp-top vial with caps for analysis, 1.8 mL.

6.11. Disposable pipettes.

6.12. Aluminum foil.

6.13. SPE Cartridges (Sep-Pak ® Vac, 6 cc (500 mg) Porapak RDX, Part No. WAT047220), or equivalent.

6.14. Vacuum manifold capable of maintaining approximately 66 cm (26") of Hg.

6.15. Concentrator tubes, 10 mL, for collecting SPE eluent

7. REAGENTS AND STANDARDS

7.1. Standards

Standards are stored at < -10°C in amber vials or ampoules. All standards are assigned an expiration date of six months from receipt or the vendor's expiration date, whichever occurs sooner. All dilutions are prepared using acetonitrile. Expiration dates for intermediate and working solutions are the same as for the parent stock standard. Standards are monitored for signs of degradation or evaporation, and replaced as necessary. Standards must be brought to room temperature before using.

7.1.1. Stock Standards

Commercial stock standards typically in the range of 100-500 ug/L are received in flame-sealed ampoules. All stocks are subject to verification of accuracy prior to use (see SOP# DEN-QA-0015 for details).

7.1.2. Intermediate Standard Solution, 10 ug/mL

A 10 ug/mL intermediate standard solution is prepared containing all analytes of interest. Preparation details may vary due to differences in commercial stocks.

7.1.3. Calibration Standards

Six calibration levels are prepared from the 10 ug/mL intermediate standard, as outlined in Table 6. Standards are prepared in a 1: 1 mixture of acetonitrile and 1% acetic acid. Additional calibration levels can be prepared at the analyst's discretion (see details concerning evaluation of calibration standards in Section 10.3).

7.1.4. Second-Source Calibration Verification Standard Solution

This standard is obtained from a different vendor than are the calibration standards described in Sections 7.1.2 and 7.1.3. A 10 ug/mL intermediate solution is prepared from stocks, which is then used to prepare a 100 ng/mL working standard used to verify the initial calibration (see Section 10.3).

7.1.5. Internal Standards

A 12.5 ug/mL solution is prepared from stocks containing 1,3-dinitrobenzene-d4, and 2,6-dinitrotoluene-d3. The internal standard solution is added to all working standards, QC and field sample extracts at 250 ng/mL. For example, if the volume of extract to be used for analysis were 1 .O mL, 20 µL of internal standard solution would be added.

7.1.6. Surrogate Standard

A 10 ug/mL solution is prepared containing nitrobenzene-d5. This standard is added to samples and QC materials prior to extraction. The concentration of surrogate standard in the final concentrated extract is 0.25 ug/L.

7.1.7. Spiking Solution, 10 ug/ml

A 10 ug/mL solution is prepared containing all anaiytes of interest. This solution is used to prepare laboratory control samples (LCSs) and matrix spike / matrix spike duplicates (MS/MSDs) at the beginning of the sample preparation procedure. Spikes are prepared at a 0.5 ug/L for waters and 25 ug/kg.

7.2. Polyethylene Glycol Tuning Solution - PEG-400,600, 800 mixture, made from neat standards obtained from a commercial source

7.3. Water - HPLC grade

7.4. Methanol -- HPLC grade

7.5. 0.01 M Ammonium Acetate - Filter through a 0.45 micron membrane filter

7.6. 1% Acetic Acid

7.7. Nitrogen gas - 99+%

7.8. Miscellaneous glassware - microsyringes, volumetric flasks, and vials for extracts

8. SAMPLE PRESERVATION AND STORAGE

8.1. Water samples should be collected in duplicate one-liter amber glass bottles with teflon lined caps. Soil samples should be collected eight-ounce wide mouth jars with teflon lined caps.

8.3. Samples and sample extracts are stored refrigerated at 4°C.

8.3. Holding times from collection to extraction are 7 days for waters and 14 days for soils. Extracts must be analyzed within 40 days from the date of extraction.

9. QUALITY CONTROL

Quality control (QC) procedures specific to this SOP are described in this section. Further quality control information, such as the process for establishing control limits, are given in QC Policy # QA-003. Any failure to meet the QC requirements listed in this section must be described in a Non-Conformance Memo (NCM), which is detailed in SOP# CORP-QA-0010.

9.1. Method Blank (MB)

A method blank (MB) is prepared and analyzed with each batch of samples. The MB consists of reagent water with the addition of surrogates and internal standards. The MB is subject to the entire extraction and analysis process. Results are acceptable if all analyte concentrations in the MB meet the following criteria:

- Results are less than or equal to the reporting limit, or
- Results are less than 1/1 0 of the measured concentration of any sample (an NCM must be prepared), or
- The same contaminants were not found in the associated samples (an NCM must be prepared).

Positive MB results slightly below the reporting limit should still be evaluated by the analyst for potential impact on sample results near the reporting limit.

Corrective Action:

If the MB does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. Samples associated with the contaminated blank shall be reprocessed for reanalysis.

9.2. Laboratory Control Sample (LCS)

A laboratory control sample (LC) is prepared and analyzed with each batch of samples. The LCS recovery must be within established control limits. The control limits shown in Table 7 will be used until sufficient data has been collected to establish three-standard-deviation control limits, as described in QC Policy # QA-003.

Corrective Actions:

If recoveries for all compounds are not within the acceptance limits, the system is out of control and corrective action must occur. Generally this requires reextraction and reanalysis of all associated samples. If the LCS is biased high and all associated samples are ND, not detected, it may be possible to report results with an NCM (see requirements for individual programs).

9.4 Matrix Spike Samples (MS/MSD)

At a minimum, the laboratory must spike one sample in every batch and spike 10% of all samples. The control limits for the spike recoveries are those shown in Table 7 until sufficient data has been collected to establish three-standard-deviation control limits, as described in QC Policy # QA-003.

Corrective Actions:

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Quality Control Check Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a duplicate LCS should be analyzed.

The matrix spike / duplicate must be analyzed at the same dilution level as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.3. Surrogates

Every sample, and QC sample is spiked with the surrogate compound nitrobenzene-d5. Surrogate recoveries are assessed to ensure that recoveries are within the 30-120% acceptance limits. Tighter control limits based on historical data may also be used. Limits are stored in the LIMS system.

Corrective Actions:

. If recoveries for surrogates in blanks or LCSs are outside of the control limits, check for, calculation or instrument problems, and reprepare and reanalyze the associated samples.

. For samples with failing surrogate recoveries, the decision to reanalyze or flag the data should be made based on agreements reached with the client or as required by the project QA plan. If matrix interference is obvious from observation of chromatograms or other objective evidence, reanalysis is unlikely to produce new or more useful information. If matrix interference is not obvious from the initial analysis, it is only necessary to reprepare and reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, so long as the extraction/instrument system is proven to be working properly.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

A mass calibration is performed monthly using the PEG solution, and is checked daily with the calibration standards. The instrument is calibrated daily with a six-point calibration curve and verified at a 10% frequency. Recommended instrument conditions are listed in Tables 2 and 3.

10.2. All standards and extracts are allowed to warm to room temperature before injecting.

10.3. Mass Calibration and Daily Mass Calibration Check

10.3.1. The PEG solution is used to perform monthly mass calibrations. The PEG is introduced directly at the APCI interface, after the HPLC. The solution is analyzed in the positive ion mode. The mass range scanned is 83 to > 1000 amu. The instrument is tuned to the masses shown in Table 8.

10.4. Initial Calibration

10.4.1. The laboratory routinely calibrates using six concentration levels (see Table 6) each day of operation, with 50 µL injections. A minimum of five calibration levels are required (six if a second order regression fit is used). The lowest point on the calibration curve is at or below the RL. See discussion in 10.4.5 regarding restrictions on rejecting initial calibration points. These concentrations defme the working range for analysis.

10.4.2. The internal standardization method is used, i.e., the ratio of the area response of the target to the area response of the nearest internal standard (see formulas in Section 12).

10.4.3. Calibrations are modeled either as average response factors or as calibration curves. When average response factors are used, the RSD must be <15%. Alternatively, a calibration curve may be used.

10.4.4. The correlation coefficient must be $\geq$ 0.990. For second-order regression fits, the coefficient of determination must by $\geq$ 0.990. While second-order regression equations may be used, the intercept and degree of curvature should be examined to be sure that results will be reliable throughout the working range. A $1/X$ or $1/X^2$ weighting is recommended.

Note about weighting:
In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/Concentration^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.45. Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or the linear range is adjusted accordingly. The only exception is that a level may be removed from the calibration if the reason can be clearly documented, for example a broken vial. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without

any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.5. Second-Source Initial Calibration Verification (ICV)

A 100 ug/L standard obtained from a different source than the initial calibration standards is used to verify the initial calibration. The result for ICV standard must be within ± 30% of the expected value.

Corrective Actions:

If this is not achieved, the calibration standards and instrument operating conditions should be checked and the instrument recalibrated.

10.6. Continuing Calibration Verification (CCV)

10.6.1. A 100 ug/L from the same source as the initial calibration standards is analyzed after every 10 samples. The CCV results must be within ± 30% difference of the expected value for each compound.

Corrective Actions:

If this is not achieved, the calibration standards and instrument operating conditions should be checked and the instrument recalibrated.

10.6.2. The internal standard (IS) response of continuing calibration standards must be within 50-200% of the response in the mid-level of the initial calibration standard,

Corrective Actions:

If this is not achieved, the chromatographic system must be inspected for malfunctions and corrected. Reanalyze any samples tested after the failing IS, prior to recalibration.

10.7. Calibration Blanks (ICB and CCB)

A solvent blank is analyzed after the ICV and after each CCV. The results must be less than the reporting limit (RL) concentration.

Corrective Actions:

If any blanks are greater than the RL, check for possible carry-over from high level samples, check cleanliness of the solvent, perform any necessary cleaning, recalibrate, and rerun samples tested since the last successful blank,

11. PROCEDURE – SAMPLE PREPARATION

11.1. Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Solid Phase Extraction of Aqueous Samples

11.2.1. Measure 1000 mL of sample. Add surrogate spikes as indicated in Appendix III.

11.2.2. Precondition the column with 10 mL of acetonitrile.

11.2.3. Condition the column with 30 mL of deionized water.

I 1.2.4. Begin dripping the sample through the cartridge at a rate of 10 mL/min. Do not prefilter the sample. Do not let the extraction tube go completely dry. If the tube clogs after sample loading, measure the amount of sample successfully extracted and use that volume for the extraction constant. A tube is considered clogged if a flow rate of 4 mL/min cannot be achieved.

11.25. If the tube is not clogged, rinse the cartridge with 10 mL of deionized water. Maintain the vacuum for about 30 seconds to assure that the cartridge is completely dry.

1 1.2.6. Shut off the vacuum and dry the vacuum manifold.

1 1.2.7. Measure 5.0 mL of deionized water into all extraction vials, mark the liquid level on the bottle and dispose of the water.

11.2.8. Put the sample collection vials in the rack and slowly (approximately one drop / 5 seconds) elute with 2.5 mL of acetonitrile by pulsing the pump once and allowing the acetonitrile to be pulled by gravity (this should take several minutes).

11.2.9. Add 2.5 mL of HPLC water to each sample.

11.2.1 O.Dilute with acetonitrile to the 5.0 mL mark on the extraction vial.

11.3, Sonication of Soil Samples

11.3.1. Sample homogenization

Weigh a minimum of 20 g of sample and dry in a forced-air oven at room temperature for 18 hours. Record the start and stop times on the benchsheet. Add the dry soil to a clean mortar and grind thoroughly with a pestle. If the sample is not completely dry, repeat the drying and grinding steps. Use Ottawa sand for method blanks and LCSs. Place the ground sample in a 30-mesh sieve. Collect all soil that passes through the sieve and place in a labeled 40 mL screw-top vial.

11.3.2. Sample extraction

11.3.2.1. Place a 2.0 g dry weight subsample of each soil sample in a 40 mL glass vial.

11.3.2.2. For the Method blank and the LCS, use 2.0 g of Ottawa sand.

11.3.2.3. For the Method Blank and the samples, add 9.0 mL CH_3CN and 1 .O mL of surrogate solution (5.0 µg/mL).

11.3.2.4. For the LCS and MS/MSD, add 8.0 mL CH_3CN, 1 .O mL of spike solution (5.0 µg/mL)*, and 1.0 mL of the surrogate solution (5.0 µg/mL).

*Note: Nitroglycerin and PETN are run at 10 times this level.

11.3.2.5. Cap vial with Teflon-lined cap, hand-shake via1 for one minute, and place in a cooled ultrasonic bath for 18 hours. Record the start time on the benchsheet.

11.3.2.6. After sonication, record the stop time and centrifuge at 4000 rpm for 5 minutes. Remove 5 mL of supernatant, and combine with 5 mL of calcium chloride solution in a labeled 10 mL amber vial. Shake for 2 minutes and let it settle for 15 minutes prior to analysis.

11.3.3. Filtering of extracts

All extracts are filtered through 0.2 µm Teflon filters into 1.8 mL screw-top vials prior to analysis.

12. PROCEDURE – INSTRUMENTAL ANALYSIS

12.1. Calibrate the instrument as described in Section 10.

12.2. All samples must be analyzed using the same instrument conditions as the calibration standards.

12.3. Add internal standard at 250 ng per mL of extract (for example, 25 µL internal standard solution in 1 .0 mL of extract) to the sample extract and mix thoroughly before injection into the instrument.

12.4. Inject 50 µL of the sample extract into the LC/MS system using the same injection technique as used for the standards.

12.5. The data system will determine the concentration of the analytes in the extract using calculations equivalent to those in section 12.

12.6. Dilutions

If the response-for any compound exceeds the working range of the LC/MS system, a dilution of the extract is prepared and analyzed. Ideally the dilution will produce an instrument response at the mid-level calibration standard or higher. The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12.7. Retention time criteria for samples

If the retention time for the internal standard changes by more than 0.33 minutes (20 seconds) from the last initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

12.8. Troubleshooting Guide

12.8.1. Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the STL Denver LQM, the following daily maintenance should be performed.

12.8.2. Major Maintenance

12.8.2.1. If minor maintenance does not result in an acceptable chromatography, it may be necessary to replace the column.

12.8.2.2. A multiplier gain check is performed if sensitivity is poor and/or analyst suspects that the multiplier is going bad.

12.8.2.3. Mass calibration is performed if the analyst notices mass assignment errors.

12.8.2.4. Refer to the manufacturer's manual for specific guidance.

13. DATA ANALYSIS AND CALCULATIONS

13.1. Qualitative identification

An analyte is identified by retention time and ion mass (see Table 4). The sample component retention time must compare to within ± 0.33 min. of the retention time of the standard component in the initial calibration. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

13.2. Calculations for Samples

13.2.1. Percent Relative Standard Deviation for initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

$\overline{RF}$ = Mean of RFs from intial caibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

RFi = RF for each of the calibration levels

N = Number of RF values

13.2.2. Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

13.2.3. Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

13.2.3.1. Average response factor

If the average of all the RSDs of the response factors in the initial calibration is $\leq 15\%$, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

13.2.3.2. Linear fit

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

C_{ex}= Concentration in extract, µg/mL

R_x= Response for analyte

R_{is}= Response for internal standard

C_{is}= Concentration of internal standard

A= Intercept

B= Slope

13.2.3.3. Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

C= Curvature

13 .2.4. The concentration in the sample is then calculated.

13.2.4.1. Aqueous Calculation

$$\text{Concentration, } \mu g / L = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a l-to-10 dilution of a 1 mL extract will mean V, = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)

V_O = Volume of water extracted (mL)

13.2.5. Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis:

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

W_S = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

13.3. LCS Percent Recovery

$$\text{Control Spike recovery} = \frac{SSR}{SA} \times 100$$

Where:

SSR = Spike sample result

SA = Spike added

13.4. MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

S_{SR} = Spike sample result

S_R=Sample result

S_A = Spike added

13.5. Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

RPD = Relative percent difference

MS_R = Matrix spike result

$MSDR$ = Matrix spike duplicate result

13.6. Reporting limits are shown in Table 1. If samples require dilutions or smaller volumes than normally used, the RL will be elevated.

13.7. All results are subject to two levels of technical review, which is documented on the checklist shown in Figure 2.

14. METHOD PERFORMANCE

Analysts performing this procedure must be skilled in the interpretation of liquid chromatograms and mass spectra. The following studies must be completed initially before samples are analyzed, and must be repeated on an annual basis.

14.1. Initial Demonstration of Capability (IDOC)

Each analyst performing this procedure must successfully analyze four LCS QC samples using current laboratory LCS control limits. The results of the IDOC study are summarized in the NELAC format, as described in SOP# DEN-QA-0024. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

14.2. Method Detection Limit (MDL)

MDLs are performed following the 40CFR136B protocol. See Policy # QA-005 for details. MDL study results are reviewed and approved by the QA staff, and are stored in QuanTIMS, the laboratory LIMS system.

15. POLLUTION PREVENTION

Solid phase extraction (SPE) minimizes solvent usage.

16. WASTE MANAGEMENT

Waste generated during preparation and from unused extracts must be disposed of in accordance with the facility hazardous waste procedures. The Health and Safety Coordinator should be contacted if additional information is required.

17. REFERENCES

SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996:

17.1. Method 353 5, for SPE extraction of waters

17.2. Method 8330, for sonication extraction of soils

17.3. Method 832 1 A, for LC/MS analysis

18. MISCELLANEOUS -

18.1. Deviations from Method 8321A

18.1.1. Method 8321A is a general purpose LC/MS or LC/UV environmental method listing a variety of instrument configurations to analyze for a wide variety of classes of compounds. Section 1.2 of the method states that "This method may be applicable to the analysis of other non-volatile or semivolatile compounds." This SOP is for analysis of a specific set of compounds, nitroaromatic explosives, using a specific LC/MS configuration (APCI and the negative ion mode). Many of the details in the method for herbicides, pesticides, and dyes do not apply to explosives analysis.

18.1.2. Tables 7 and 8 of the method list calibration masses for PEG 400 and PEG 600, which are generally different than the masses listed in Table 9 of this SOP. The ion abundances are all 100% for this procedure, which is also different than the Tables in the method. This is because, the text of 832 1A includes PEG 800, but there is no Table of calibration masses in the method. Table 9 of this SOP includes calibration masses for all three forms of PEG.

18.2. Changes from Revision 1

18.2.1. Full details about the preparation were added, rather than referring to the 8330 SOP.

18.2.2. The reporting limits for soils were adjusted downward based on recent best available information.

18.2.3. Instrument conditions are changed to allow the separation of 2-nitrotoluene and 4-nitrotoluene. We now have separation for all target analytes.

Table 1
Analyte Reporting Limits

Compound	CAS Number	Reporting Limit Soil (ug/kg)	Reporting Limit Water (ug/L)
HMX	2691-41-0	250	0.12
RDX	121-82-4	250	0.12
1,3,5-Trinitrobenzene	99-35-4	250	0.12
1,3-Dinitrobenzene	99-65-0	250	0.12
Tetryl	479-45-8	250	0.12
2,4,6-Trinitrotoluene	118-96-7	250	0.12
Nitrobenzene	98-95-3	250	0.12
Nitroglycerin	55-63-0	250	0.12
2,4-Dinitrotoluene	121-14-2	250	0.12
2-Amino-4,6-dinitrotoluene	355-72-78-2	250	0.12
2,6-Dinitrotoluene	606-20-2	250	0.12
4-Amino-2,6-dinitrotoluene	1946-51-0	250	0.12
2-Nitrotoluene	88-72-2	250	0.12
4-Nitrotoluene	99-99-0	250	0.12
3-Nitrotoluene	99-08-1	250	0.12
PETN	78-11-5	250	0.12

Table 2
HPLC Operating Conditions

Parameter	Setting
Column	250 X 3.2 mm C_{18}; AllTech Adsorbosphere HS 5µ or equivalent
Column Temperature	25°C
Eluant Flow Rate	0.25 mL/min
Eluant Identification	A = Water (0.01M NH_4Ac) B = Methanol (0.01M NH_4Ac)
Solvent Program	40% A + 60% B Isocratic, 30 min. run time
Injection Volume	50 µL

Table 3
Recommended Mass Spectrometer Operating Conditions

Parameter	Setting
Scan Mode	APCI, Negative Ion
Corona Current	30 μamps
Multiplier Voltage	650 Volts
Vaporizer Temperature	400°C
Source Temperature	120°C
Desolvation Gas Flow	400 L/hr
Cone Gas Flow	120 L/hr

Table 4
Characteristic Ions and Scan Conditions
Compounds in approximate retention time order

Analyte	Parent Ion (m/z)	Dwell Time (sec)
HMX	355	0.5
RDX	281	0.5
1,3,5-Trinitrobenzene	212.9	0.4
1,3-Dinitrobenzene-d4 (Internal standard)	172	0.4
1,3-Dinitrobenzene	168	0.4
Tetryl	241	0.4
2,4,6-Trinitrotoluene	226.9	0.4
Nitrobenzene-d5 (Surrogate)	128	0.4
Nitrobenzene	123	0.4
Nitroglycerin	241	0.4
2,4-Dinitrotoluene	182	0.4
2-Amino-4,6-dinitrotoluene	197	0.4
2,4-Dinitrotoluene-d3 (Internal standard)	185	0.4
2,6-Dinitrotoluene	182	0.4
4-Amino-2,6-dinitrotoluene	197	0.4
2-Nitrotoluene	136.9	0.4
4-Nitrotoluene	136.9	0.4
3-Nitrotoluene	136.9	0.4
PETN	378	0.6

Table 5
Internal standard and Surrogate Recovery Limits

Compounds	% Recovery
1,3-Dinitrobenzene-d4 (IS)	50-200
2,6-Dinitrotoluene-d3 (IS)	50-200
Nitrobenzene-d5 (Surr)	30-120

Table 6
Calibration Levels, ng/mL

Analytes	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
All Analytes	10	25	50	100	200	300
Internal standards	250	250	250	250	250	250

Calibration solutions are prepared in a 1:1 solution of acetonitrile and 1% acetic acid.

Table 7
Initial Demonstration recovery and precision limits

Analyte	Spiking Level ug/L	Limit for %RSD	Limit for average recovery, %
All Analytes	0.5	30	30-120

Table 8 – Calibration Masses for PEG Solution (amu)
80.07
124.09
168.12
212.15
256.18
300.20
344.23
388.25
432.28
476.30
520.33
564.36
608.38
652.41
696.44
740.46
784.49
828.52
872.54
916.57
960.57
1004.62

All ionabundancesare 100%.

Figure 1: SPE Extraction Flow Chart

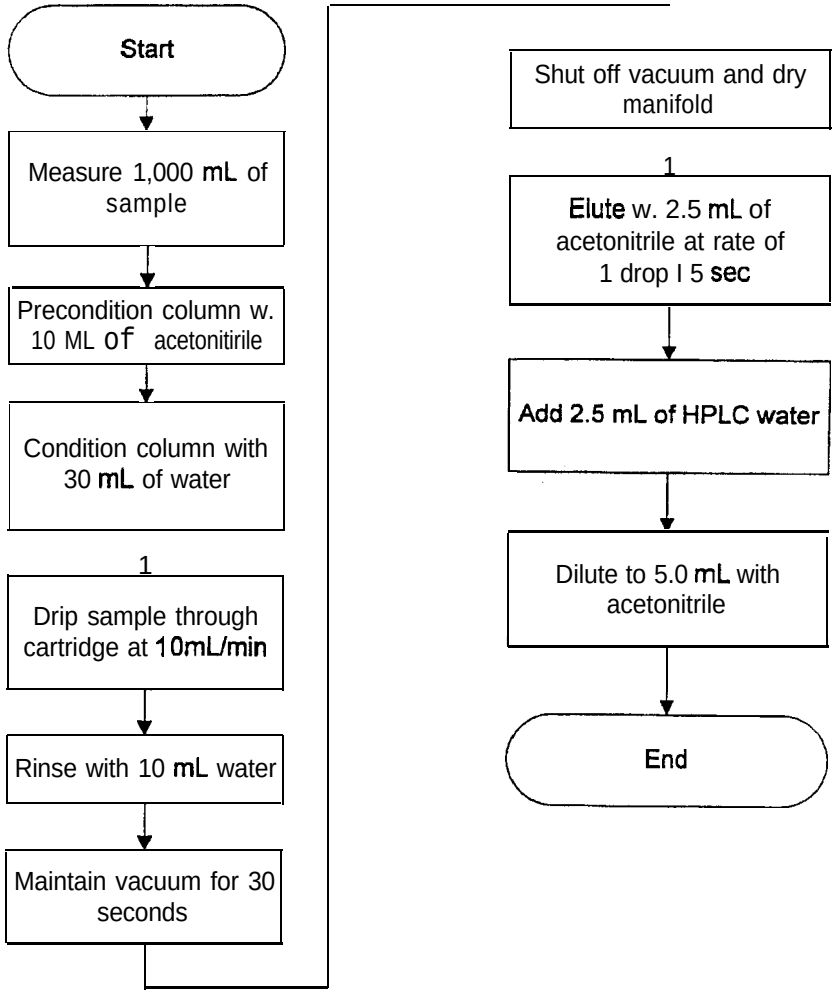


Figure 2: Soil Extraction Flow Chart

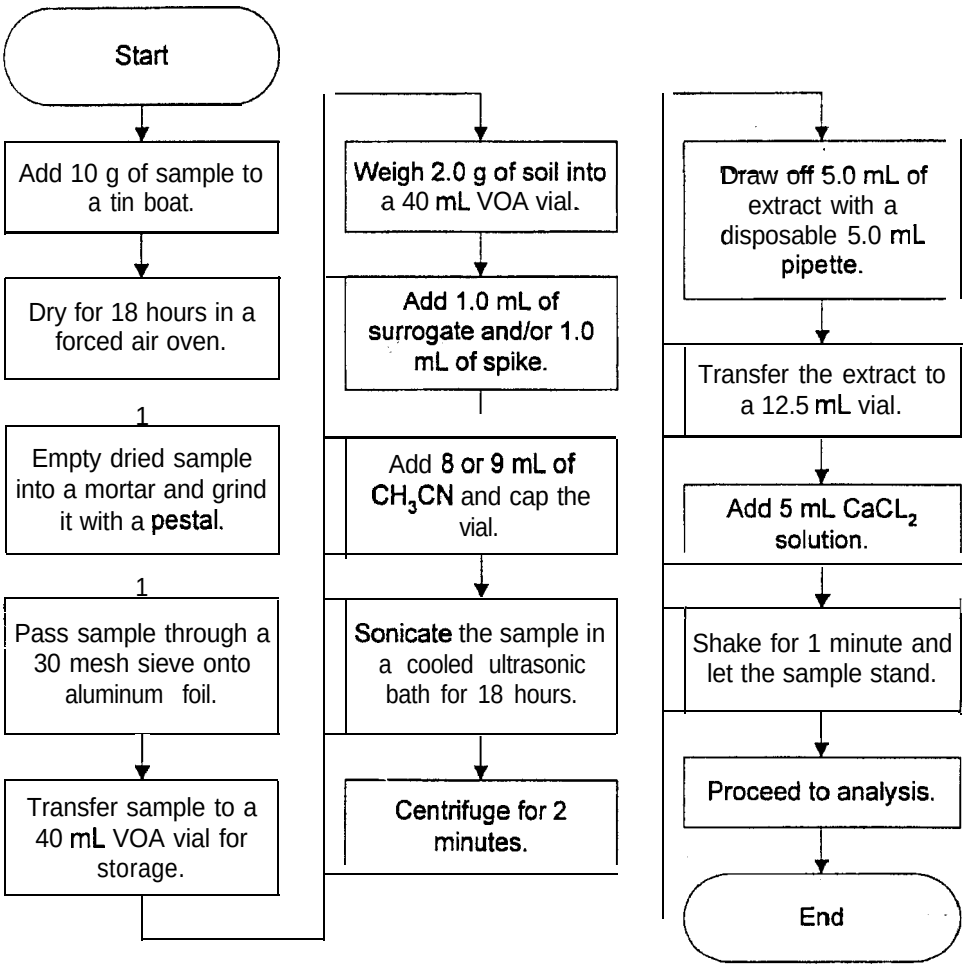


Figure 3: LC/MS Analysis

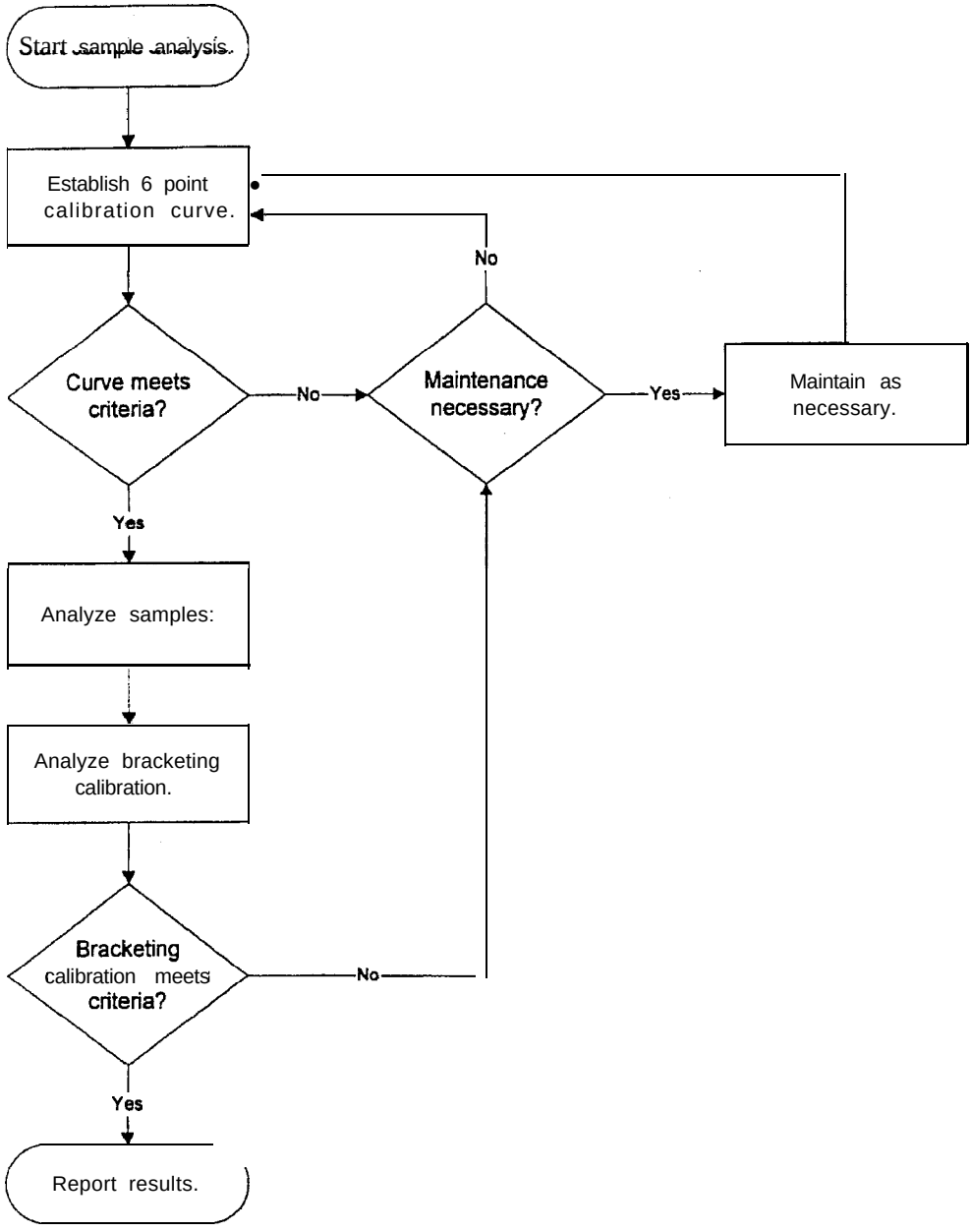


Figure 4: Data Review Checklist

STL Denver -- LC/MS Data Review Checklist

Review Items	Level 1 Yes	Level 1 No	Level 1 N/A	Level 2	Comments & Samples Affected
Instrument					
1. Was instrument check performed?					
Initial Calibration					
1. ICAL date verified?					
2. Sufficient number of calibration points used?					
3. Reasons for removal of points documented?					
4. %RSD or correlation coefficient within acceptable limits?					
5. Isometric pairs checked for correct peak assignment?					
6. Manual integrations documented and checked?					
Client Samples & QC Sample Results					
1. Was the correct analysis performed & project instructions followed?					
2. Was correct SOP and protocol followed?					
3. Was preparation and analysis done within holding times?					
4. Are samples quantitated against correct RF or calibration curve?					
5. Were spectra for positive results evaluated correctly?					
6. Are positive results within calibration range?					
7. Dilutions due to target cpds? ____ Dilutions due to non-targets? ____					
8. Are internal standard (IS) recoveries acceptable?					
9. Are target constitients and surrogates in LCS/DCS acceptable?					
10. Are target cpds in fortified placebo (method blank) < RL? (requires NCM if "no")					
11. Are fortified (surrogate) recoveries for fortified placebo acceptable?					
12. Are fortified sample (MS/MSD) recoveries and precision (RPDs) acceptable?					
13. Manual integrations documented and checked?					
14. Are nonconformances documented on an NCM?					
15. Is the appropriate raw data included?					
16. Are all results manually entered into LIMS verified by 2nd person?					

1st Level Reviewer: _____ Date: _____

2nd Level Reviewer: _____ Date: _____

Severn Trent Laboratories, Applied Analytical Services Group

METHOD DETECTION LIMIT STUDY (Aqueous)

DATE COMPLETED:	1/9/01	INSTRUMENT ID:	MicroMass
METHOD NUMBER:	8321	LOT NUMBER:	Run File exp0108, exp0110
METHOD DESCRIPTION:	LCMS Explosives	ANALYST:	Christopher Borton
PREP METHOD:	SPE, no salt-out	QUALITY ASSURANCE:	

1

ANALYTE	SPIKE CONC ug/L	REPLICATE MEASUREMENT 1	2	3	4	5	6	7	AVG ug/L	Recovery of Spike %	PREC. ug/L	MDL ug/L	Report. Limit ug/L
HMX (exp0108)	0.075	0.095	0.092	0.093	0.091	0.094	0.097	0.096					
HMX (exp0110)	0.075	0.112	0.111	0.107	0.108	0.107	0.108	0.111	0.102	135.43%	0.00812	0.022	0.12
RDX (exp0108)	0.075	0.113	0.113	0.116	0.108	0.115	0.115	0.117					
RDX (exp0110)	0.075	0.136	0.134	0.130	0.133	0.132	0.133	0.138	0.124	165.05%	0.0106	0.028	0.12
1,3,5-Trinitrobenzene (exp0108)	0.075	0.088	0.077	0.092	0.088	0.093	0.101	0.096					
1,3,5-Trinitrobenzene (exp0110)	0.075	0.088	0.080	0.085	0.087	0.083	0.082	0.089	0.088	117.05%	0.00639	0.017	0.12
1,3-Dinitrobenzene (exp0108)	0.075	0.110	0.097	0.102	0.113	0.098	0.096	0.099					
1,3-Dinitrobenzene (exp0110)	0.075	0.111	0.095	0.098	0.116	0.100	0.091	0.098	0.102	135.62%	0.00761	0.020	0.12
Tetryl (exp0108)	0.075	0.062	0.071	0.068	0.074	0.052	0.064	0.058					
Tetryl (exp0110)	0.075	0.064	0.062	0.062	0.067	0.048	0.054	0.058	0.062	82.29%	0.00726	0.019	0.12
2,4,6-Trinitrotoluene (exp0108)	0.075	0.093	0.088	0.088	0.097	0.101	0.094	0.099					
2,4,6-Trinitrotoluene (exp0110)	0.075	0.060	0.057	0.057	0.062	0.057	0.057	0.066	0.077	102.48%	0.0186	0.049	0.12
Nitrobenzene (exp0108)	0.075	0.072	0.077	0.077	0.070	0.072	0.075	0.077					
Nitrobenzene (exp0110)	0.075	0.051	0.064	0.063	0.053	0.055	0.064	0.057	0.066	88.29%	0.00938	0.025	0.12
2,4-Dinitrotoluene (exp0108)	0.075	0.088	0.078	0.081	0.084	0.083	0.083	0.085					
2,4-Dinitrotoluene (exp0110)	0.075	0.078	0.068	0.070	0.075	0.072	0.073	0.076	0.078	104.19%	0.00609	0.016	0.12
2-Amino-4,6-dinitrotoluene (exp0108)	0.075	0.084	0.077	0.078	0.078	0.082	0.079	0.083					
2-Amino-4,6-dinitrotoluene (exp0110)	0.075	0.076	0.068	0.070	0.073	0.073	0.073	0.078	0.077	102.10%	0.00475	0.013	0.12
2,6-Dinitrotoluene (exp0108)	0.075	0.082	0.074	0.079	0.081	0.081	0.080	0.082					
2,6-Dinitrotoluene (exp0110)	0.075	0.079	0.068	0.070	0.074	0.074	0.074	0.075	0.077	102.19%	0.00453	0.012	0.12
4-Amino-2,6-dinitrotoluene (exp0108)	0.075	0.085	0.079	0.079	0.079	0.081	0.079	0.085					
4-Amino-2,6-dinitrotoluene (exp0110)	0.075	0.072	0.067	0.068	0.068	0.071	0.070	0.076	0.076	100.86%	0.00625	0.017	0.12
2 & 4-Nitrotoluene (exp0108)	0.150	0.140	0.133	0.141	0.141	0.141	0.142	0.142					
2 & 4-Nitrotoluene (exp0110)	0.150	0.107	0.116	0.115	0.110	0.117	0.127	0.108	0.127	84.76%	0.0143	0.038	0.12
3-Nitrotoluene (exp0108)	0.075	0.068	0.067	0.068	0.065	0.069	0.063	0.077					
3-Nitrotoluene (exp0110)	0.075	0.054	0.059	0.058	0.052	0.059	0.058	0.052	0.062	82.76%	0.00731	0.019	0.12
PETN (exp0108)	0.075	0.089	0.081	0.093	0.095	0.096	0.090	0.093					
PETN (exp0110)	0.075	0.084	0.083	0.077	0.072	0.078	0.078	0.078	0.085	113.14%	0.00773	0.020	0.12
Nitrobenzene-d5 (exp0108)	0.075	0.053	0.058	0.056	0.049	0.061	0.056	0.058					
Nitrobenzene-d5 (exp0110)	0.075	0.037	0.044	0.041	15	0.043	0.049	0.032	0.048	63.90%	0.00944	0.025	0.12

Severn Trent Laboratories, Applied Analytical Services Group

METHOD DETECTION LIMIT STUDY (Aqueous)

DATE COMPLETED:	1/9/01	INSTRUMENT ID:	MicroMass
METHOD NUMBER:	8321	LOT NUMBER:	Run File exp0108, exp0110
METHOD DESCRIPTION:	LCMS Explosives	ANALYST:	Christopher Borton
PREP METHOD:	SPE, no salt-out	QUALITY ASSURANCE:	

ANALYTE	SPIKE CONC ug/L	REPLICATE MEASUREMENT 1	2	3	4	5	6	7	AVG ug/L	Recovery of Spike %	PREC. ug/L	MDL ug/L	Report. Limit ug/L
Nitroglycerin (exp0108)	0.075	0.037	0.027	0.022	0.022	0.015	0.032	0.053					
Nitroglycerin (exp0110)	0.075	0.069	0.048	0.050	0.061	0.070	0.063	0.050	0.044	56.95%	0.0184	0.049	0.12

Note: The 1st seven point were obtained on 1/8/01. To get a more representative MDL, the 7 standards were analyzed again on 1110101.

A Student%-t value for 13 degrees of freedom = 2.65 was used, instead of 3.14.

STL Denver

DEMONSTRATION OF CAPABILITY STUDY

DATE COMPLETED: 12/20/00

METHOD NUMBER: 8321 A Explosives

METHOD DESCRIPTION:

PREP METHOD: Sonication

ANALYST: Chris Borton

REVIEWER / DATE: 12/20/00

COMMENTS:

QUALITY ASSURANCE: R. Johnson

ANALYTE	SPIKE CONC ug/kg	LCS RESULTS 12/20/00 1	12/20/00 2	12/20/00 3	12/20/00 4	Avg. Rec. (X%) %	Avg. (X) ug/L	Std.Dev. (s) ug/L	RSD %	Acceptance for X% lower %	upper %	for RSD %	J - - L - p & - X% $	RSD
HMX	0.5	0.357	0.365	0.417	0.408	77.4%	0.387	0.030	7.0%	30.0%	120.0%	30.0%	p	p
1,3,5-Trinitrobenzene	0.5	0.280	0.298	0.400	0.359	66.9%	0.334	0.055	16.6%	30.0%	120.0%	30.0%	p	p
RDX	0.5	0.316	0.332	0.396	0.364	70.4%	0.352	0.035	10.1%	30.0%	120.0%	30.0%	p	p
1,3-Dinitrobenzene	0.5	0.391	0.412	0.428	0.394	81.3%	0.406	0.017	4.2%	30.0%	120.0%	30.0%	p	p
Nitrobenzene	0.5	0.291	0.328	0.320	0.300	62.0%	0.310	0.017	5.5%	30.0%	120.0%	30.0%	p	p
2,4,6-Trinitrotoluene	0.5	0.364	0.427	0.390	0.366	77.4%	0.387	0.029	7.6%	30.0%	120.0%	30.0%	p	p
Tetryl	0.5	0.270	0.304	0.327	0.312	60.7%	0.303	0.024	8.0%	30.0%	120.0%	30.0%	p	p
2,4-Dintrotoluene	0.5	0.357	0.385	0.446	0.406	79.7%	0.399	0.037	9.4%	30.0%	120.0%	30.0%	p	p
2,6-Dinitrotoluene	0.5	0.398	0.415	0.425	0.423	83.1%	0.415	0.012	3.0%	30.0%	120.0%	30.0%	p	p
2-Amino-4,6-dinitrotoluene	0.5	0.376	0.397	0.397	0.359	76.5%	0.382	0.018	4.8%	30.0%	120.0%	30.0%	p	p
4-Amino-2,6-dinitrotoluene	0.5	0.353	0.357	0.417	0.375	75.1%	0.376	0.029	7.8%	30.0%	120.0%	30.0%	p	p
3-Nitrotoluene	0.5	0.312	0.356	0.336	0.329	66.8%	0.334	0.018	5.5%	30.0%	120.0%	30.0%	p	p
2&4-Nitrotoluene	1.0	0.592	0.717	0.673	0.662	66.1%	0.661	0.052	7.8%	30.0%	120.0%	30.0%	p	p

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: SONICATION Low Level
Method: Nitroaromatics & Nitramines "Explosives" (8330)
QC Program: STANDARD TEST SET
Location: STL Denver

Analyte List			Detection Limits			Check List 4139					Spike List 4139				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
4-Amino-2,6-dinitrotoluene	0.25	ug/g	0.120	ug/g	20001007	2.5	ug/g	59	134	35	2.5	ug/g	59	134	35
2-Amino-4,6-dinitrotoluene	0.25	ug/g	0.143	ug/g	20001007	2.5	ug/g	61	134	42	2.5	ug/g	61	134	42
1,3-Dinitrobenzene	0.25	ug/g	0.073	ug/g	19990202	2.5	ug/g	68	135	21	2.5	ug/g	68	135	21
2,4-Dinitrotoluene	0.25	ug/g	0.116	ug/g	20001007	2.5	ug/g	70	137	21	2.5	ug/g	70	137	21
2,6-Dinitrotoluene	0.25	ug/g	0.059	ug/g	20001007	2.5	ug/g	69	140	32	2.5	ug/g	69	140	32
HMX	0.25	ug/g	0.168	ug/g	20001007	2.5	ug/g	61	139	30	2.5	ug/g	61	139	30
Nitrobenzene	0.25	ug/g	0.128	ug/g	20001007	2.5	ug/g	61	125	24	2.5	ug/g	61	125	24
Nitroglycerin	5.0	ug/g	1.09	ug/g	20001007										
2-Nitrotoluene	0.25	ug/g	0.196	ug/g	20001007	2.5	ug/g	60	131	26	2.5	ug/g	60	131	26
3-Nitrotoluene	0.25	ug/g	0.152	ug/g	20001007	2.5	ug/g	56	134	23	2.5	ug/g	56	134	23
4-Nitrotoluene	0.25	ug/g	0.137	ug/g	20001007	2.5	ug/g	65	129	26	2.5	ug/g	65	129	26
PETN	2.5	ug/g	1.01	ug/g	20001007	5.0	ug/g	50	150	20	5.0	ug/g	50	150	20
RDX	0.25	ug/g	0.215	ug/g	20001007	2.5	ug/g	73	123	25	2.5	ug/g	73	123	25
Tetryl	0.50	ug/g	0.183	ug/g	20001007	2.5	ug/g	44	116	38	2.5	ug/g	44	116	38
1,3,5-Trinitrobenzene	0.25	ug/g	0.125	ug/g	19990202	2.5	ug/g	48	135	33	2.5	ug/g	48	135	33
2,4,6-Trinitrotoluene	0.25	ug/g	0.111	ug/g	20001007	2.5	ug/g	61	129	26	2.5	ug/g	61	129	26
1,2-Dinitrobenzene						2.5	ug/g	74	144	0	2.5	ug/g	74	144	0

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: WATER
Extraction: SOLID PHASE EXTRACTION (NOMINAL)
Method: 8321A Explosives by LCMS
QC Program: STANDARD TEST SET
Location: STL AASG - Denver

Analyte List			Detection Limits			Check List 4395					Spike List 4395				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
4-Amino-2,6-dinitrotoluene	0.12	ug/L	0.017	ug/L	20010110	0.50	ug/L	48	107	30	0.50	ug/L	48	107	30
2-Amino-4,6-dinitrotoluene	0.12	ug/L	0.013	ug/L	200101 10	0.50	ug/L	47	119	30	0.50	ug/L	47	119	30
1,3-Dinitrobenzene	0.12	ug/L	0.020	ug/L	20010110	0.50	ug/L	41	138	30	0.50	ug/L	41	138	30
2,4-Dinitrotoluene	0.12	ug/L	0.016	ug/L	20010110	0.50	ug/L	53	118	30	0.50	ug/L	53	118	30
2,6-Dinitrotoluene	0.12	ug/L	0.012	ug/L	20010110	0.50	ug/L	71	113	30	0.50	ug/L	71	113	30
HMX	0.12	ug/L	0.022	ug/L	2 0 0 1 0 1 1 0	0.50	ug/L	37	132	30	0.50	ug/L	37	132	30
Nitrobenzene	0.12	ug/L	0.025	ug/L	20010110	0.50	ug/L	35	110	30	0.50	ug/L	35	110	30
Nitroglycerin	0.12	ug/L	0.049	ug/L	20010110	0.50	ug/L	25	154	30	0.50	ug/L	25	154	30
3-Nitrotoluene	0.12	ug/L	0.019	ug/L	20010110	0.50	ug/L	37	120	30	0.50	ug/L	37	120	30
2&4-Nitrotoluene	0.25	ug/L	0.038	ug/L	20010110	0.50	ug/L	39	118	30	0.50	ug/L	39	118	30
PETN	0.12	ug/L	0.020	ug/L	20010110	0.50	ug/L	34	148	30	0.50	ug/L	34	148	30
RDX	0.12	ug/L	0.028	ug/L	20010110	0.50	ug/L	25	146	30	0.50	ug/L	25'	146	30
Tetryl	0.12	ug/L	0.019	ug/L	20010110	0.50	ug/L	40	99	30	0.50	ug/L	40	99	30
1,3,5-Trinitrobenzene	0.12	ug/L	0.017	ug/L	20010110	0.50	ug/L	38	115	30	0.50	ug/L	38	115	30
2,4,6-Trinitrotoluene	0.12	ug/L	0.049	ug/L	20010110	0.50	ug/L	64	112	30	0.50	ug/L	64	112	30
Nitrobenzene-d5						0.50	ug/L	30	126	0	0.50	ug/L	30	126	0

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

THE DETERMINATION OF NITROAROMATICS AND NITRAMINES IN WATER AND SOIL BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY WITH A MASS SPECTROMETER (LC/MS) BY METHOD 8321.

(SUPERSEDES: SAC-LC-0001, REVISION 6.0)

Prepared by: Dennis E. Gall

Reviewed by: _____
Technical Specialist, Steven D. Rogers

Approved by: _____
Quality Assurance Manager, Pamela Schemmer

Approved by: _____
Environmental Health and Safety Coordinator, Joe Schairer

Approved by: _____
Laboratory Manager, Eric Redman

1. SCOPE AND APPLICATION

1.1. This method is suitable for the extraction and analysis of explosive residues listed below in water and in soil and sediment by High Performance Liquid Chromatography with a Triple-Stage Quadrapole Mass Spectrometer (LC/MS) using an atmospheric pressure chemical ionization (APCI) interface.

Test Components	Abbreviation	CAS Number
2-Amino-4,6-dinitrotoluene	2-AM	35572-78-2
4-Amino-2,6-dinitrotoluene	4-AM	1946-51-0
1,3-Dinitrobenzene	DNB	99-65-0
2,4-Dinitrotoluene	2,4-DNT	121-14-2
2,6-Dinitrotoluene	2,6-DNT	606-20-2
Hexahydro-1,3,5-trinitro-1,3,5-triazine (Hexogen)	RDX	121-82-4
Methyl-2,4,6-trinitrophenylnitramine	TETRYL	479-45-8
Nitrobenzene	NB	98-95-3
2-Nitrotoluene (o-Nitrotoluene)	2-NT	88-72-2
3-Nitrotoluene (m-Nitrotoluene)	3-NT	99-08-1
4-Nitrotoluene (p-Nitrotoluene)	4-NT	99-99-o
Octahydro-1,3,5,7-tetranitro1,3,5,7-tetracine (Octogen)	HMX	2691-41-0
1,3,5-Trinitrobenzene	TNB	99-35-4
2,4,6-Trinitrotoluene	TNT	118-96-7
Glycerol Trinitrate (Nitroglycerin)	NG	55-63-0
Pentaerythritol Tetranitrate	PETN	78-11-5

1.2. This method is NOT applicable for high concentrations of explosives in soil or waste samples. Method 8515 should be used to determine whether high concentrations of explosives are present in soil or waste samples.

1.3. The standard range for most 8321 compounds is 10 ng/mL to 1,000 ng/mL with the exception of nitroglycerin and PETN is 100 ng/mL to 8,000 ng/mL. The standard is prepared in 25/75 (v/v) acetonitrile (containing 0.1% acetic acid)/water.

1.4. With the addition of acetic acid in the extracting solvent and in standard solution, TETRYL has been stable for months without affecting the other nitroaromatics and nitramines.

1.5. Aqueous samples are extracted by Solid Phase Extraction (SPE).

1.6. Soil samples and wipe samples are extracted with 0.1% acetic acid in acetonitrile by sonication.

2. SUMMARY

2.1. Aqueous samples are extracted by solid phase extraction (SPE). The samples are extracted within 7 days of sampling and analyzed within 40 days from the date of extraction.

2.1.1. In the SPE method, aqueous samples are extracted by passing through Porapak RDX cartridges, where the components are absorbed to and eluted from with 0.1% acetic acid in acetonitrile. The eluants are filtered and diluted 4× with water, and internal standards are added and analyzed by LC/MS.

2.2. Soil and sediment samples that appear to be non-homogeneous and are not suspicious are dried, ground to a finer texture, and sieved through a #30 mesh screen. A sub sample of the dried, finely grind soil is extracted with 0.1% acetic acid in acetonitrile by sonication in a temperature-controlled water bath for 18 hours. An aliquot is diluted 4× with water, filtered, and internal standards are added and analyzed by LC/MS.

2.3. Acetic acid at 0.1% volume in acetonitrile improves the stability of TETRYL.

2.4. The extracts are analyzed on a C_{18} reverse phase column, such as Carbosorb ODS-2 by LC/MS with an APCI interface.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Manual (LQM).

4. INTERFERENCES

4.1. The solvents, reagents, glassware, and other sample process hardware used in the extraction and analysis of nitroaromatics, nitramines, and specialty explosives must free of interference. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running method blanks.

4.2. Glassware must be cleaned, dried, and solvent rinsed to maximize cleanliness and minimize interferences.

4.3. HPLC and pesticide grade reagents must be used to minimize interference problems.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.2.1. Neoprene, natural rubber, and butyl gloves provide varying degrees of protection against those chemicals listed. Refer to permeation/degradation charts for the actual data.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA includes: methylene chloride(used primarily in the rinsing of glassware).

5.3.2. Chemicals known to be flammable in this method are acetone, acetonitrile, and methanol.

5.3.3. The following materials are known to be corrosive: acetic acid.

5.3.4. Hearing protection may be recommended when ultrasonic digestion is carried out.

5.4. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.5.1. Explosive standards in this method are secondary explosives. As neat, they are by nature unstable materials and they must be stored according to manufacturer's directions, preferably wet. However, if the standards are not

purchased as neat but as solutions at low concentration in organic solvents, safety hazards are minimized.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of an associate. The situation must be reported immediately to a laboratory supervisor.

5.7. Each chemical compound must be treated as a potential health hazard, especially when the components are considered extremely explosive and should be kept wet in small quantities. All the compounds in this method are either used in the manufacturing of explosives or are the degradation products of compounds used in the manufacturing of explosives. Treat each compound as if it were extremely explosive when handling and preparing stock solutions for calibration.

5.8. Soil samples having as high as 2% TNT can be safely ground. Samples containing higher levels should not be ground. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.

5.9. Soil samples may be screened or tested before grinding if they are suspected to contain high levels of explosives using the TNT/RDX Soil Extraction PAC®. Follow the manufacturer's instructions.

6. EQUIPMENT AND SUPPLIES

6.1. Balances - Top loading, capable of accurately weighing to the nearest 0.01 gram. Analytical, capable of accurately weighing to the nearest 0.0001 gram.

6.2. Centrifuge.

6.3. Filter assembly, 25-mm, 0.45 um pore size PTFE filters, such as Acrodisc 25.

6.4. Liquid chromatograph.

6.4.1. LC/MS - Liquid Chromatography/Thermospray/Mass Spectrometer. An analytical system completed with sample injection loop, analytical columns, a triple-stage quadrapole mass spectrometer, and data system.

6.5. Mesh sieve and pan, brass or stainless steel, mesh size # 30.

6.6. Mortar and pestle, ceramic.

6.6.1. Grinder, Retch model #RM 100, with ceramic bowl and pestle, or equivalent.

6.6.2. Waring Blender, stainless steel container, and top in place of the motar and pestle is acceptable if samples are homogenized and capable of passing through a wired #30 mesh sieve.

6.7. Re-circulating cooler.

6.8. Spatula, stainless steel, or equivalent.

6.9. Syringe, 3-cc and 10-cc disposable.

6.10. Temperature controlled ultrasonic water bath.

6.11. Test tubes, 8-mL and 16-mL, glass with teflon-lined screw cap.

6.12. Vials, 40-mL, glass with teflon-lined screw cap.

6.13. Workstation, Zymark SPE AutoTrace, an automated workstation to perform solid phase extraction on aqueous samples.

7. SOLVENTS, REAGENTS AND STANDARDS

NOTE: All preparations of reagents and solutions are entered in the Reagent Prep Notebook.

7.1. Acetic acid, Glacial, Reagent grade.

7.1.1. Acetic acid in acetonitrile, 0.1% v/v.

7.2. Acetone, pesticide quality.

7.3. Acetonitrile, HPLC grade.

7.4. Ammonium Acetate, HPLC grade.

7.4.1. Ammonium acetate in water, 1.0 M. Weigh 77.5 grams of ammonium acetate into 1,000 mL of water.

7.4.2. Ammonium acetate in water, 0.01 M. Dilute 10.0 mL of 1.0 M Ammonium acetate solution in 990 mL of water.

7.5. Methanol, HPLC grade.

7.5.1. Ammonium acetate in methanol, 0.01 M. Dilute 10.0 mL of 1.0 M Ammonium acetate solution in 990 mL of methanol.

7.6. Water, nanopure and HPLC grade.

7.7. Standards may be prepared from neat or purchased as certified solutions. All standard preparations must be entered in the standards prep log.

7.7.1. 8321 Mix -A prepared stock mix of the standards at 1.0 mg/mL in acetonitrile/methanol (1:1) is used for the preparation of fortification and analytical solutions. They are obtained from EM Science, Accustandard, Ultra Scientific, Radian International, or other reputable source. The concentration of the stock mix solution is 1.0 mg/mL, and expires in approximately one year if properly stored. Once the ampule is broken and the unused portion transferred to an amber vial, sealed with teflon-lined screw cap, and stored in the freezer at -10°C or less, this solution is valid for 30 days.

7 . 7 2 Prepare primary 8321mix solution at 50 ug/mL and 5.0 ug/mL, each in 0.1% acetic acid in acetonitrile from the stock mix. Store the solutions in the freezer in the dark and at -10°C or less, and replace after 30 days. These solutions are used for fortification of samples, and the preparation of the analytical standards.

7.8. Standards must be reviewed for expiration dates at least monthly. All expired standards must be rotated out of the laboratory to the Haz-Waste storage area for disposal per the CHP.

7.9. SPECIALTY EXPLOSIVES (SPEX) – Nitroglycerin and PETN are received as solutions at 1.0 mg/mL in ethanol, methanol, or acetonitrile (obtained from Radian International, EM Science, Accustandard, Ultra Scientific, or other reputable source).

7.9.1. A mix solution of nitroglycerin and PETN (NG/PETN) is prepared at 50 ug/mL in acetonitrile. Store the solution in the refrigerator at 4 ± 2°C and replace the solution after 6 months. This solution is also used in the fortification of samples.

7.10. Surrogates

7.10.1. Method 8321-EXP

- Nitrobenzene-d_5 / NB-d_5 / CAS No. 4165-60-0.

7.10.2. Prepare an individual stock solution from neat (obtained from CIL) at approximately 1.0 mg/mL in acetonitrile. Store the stock solution in the refrigerator and replace the solution after 1 year.

7.10.3. Prepare the surrogate solution from the stock solution at 50 ug/mL in acetonitrile. Store the solution in the freezer and replace every six months.

7.11. Internal Standards for LC/TSP/MS;

- RDX-$^{13}C_3$ / CAS No. 121-82-4.
- 2,6-Dinitrotoluene-d_3 / CAS No. 606-20-2.
- 1,3-dinitrobenzene-d_4 / CAS No. 99-65-0.

7.11.1. Prepare an 8321-EXP-IS solution (containing RDX-$^{13}C_3$, 2,6-dinitrotoluene-d_3, and 1,3-dinitrobenzene-d_4) at 10 ug/mL in acetonitrile from the individual stock solutions or from solutions purchased from CIL or other reputable source. Store the solution in the refrigerator at 2-6°C and replace after 6 months.

7.12. Analytical Curve.

7.12.1. The 8321 and specialty explosives (SPEX) analytical curve is prepared at the following concentrations listed in table 6 below in 25:75 acetonitrile (containing 0.1% acetic acid):water.

Levels, in **ng/mL**	**1**	**2**	3	4	**5**	6
8321 Mix	10	25	100	200	500	1000
Nitroglycerin	100	200	1000	2000	4000	8000
PETN	100	200	1000	2000	4000	8000
Surr; NB-d_5	20	50	100	150	200	250

* where the 8321 mix is the 14 standard components.

7.12.2. A 4.0 mL portion of each standard is transferred to a 4-cc vial, and 20 uL of the 8321-EXP-IS is added.

Levels, in ng/mL	1	2	3	4	5	6
IS1; RDX-$^{13}C_3$	50	50	50	50	50	50
IS2; DNT-d_3	50	50	50	50	50	50
IS3; DNB-d_4	50	50	50	50	50	50

7.13. Reference Standards.

7.13.1. Prepare reference standards to verify the quality of the fortification solutions and analytical standards above.

7.13.2. A reference standard is any standard solution made from a source other than the stock standard. Reference standards may be from the EPA, the manufacturer, or from another reliable source.

7.13.3. If a secondary source is not available, a separate intermediate stock solution will be made from the same neat by another chemist or from a neat from a different vendor and different lot number.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Aqueous samples are collected in glass bottles, sealed with teflon-lined screw caps and iced or refrigerated at 4 ± 2°C and protected from sunlight from time of collection until extraction.

8.2. The extraction hold time for aqueous samples is 7 days from the time of sampling to extraction and analyzed within 40 days from extraction.

8.3. Soil samples are collected in glass jars with teflon-lined screw caps and iced or refrigerated at 4 ± 2°C and covered from sunlight from time of collection until extraction.

8.4. Soil samples are to be air dried, finely ground and sifted, and extracted within 14 days of sampling and analyzed within 40 days of extraction.

8.5. Soil and sediment samples should be air dried at room temperature until there is no visible appearance of moisture. Successful grinding and sifting verifies dryness. While it is possible to analyze wet soil samples, it is much more difficult to obtain a homogeneous sub-sample on a wet sample. If wet soil samples are to be analyzed, a moisture determination must be made on a separate sub-sample.

9. QUALITY CONTROL

9.1. One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples the method blank is an aliquot of laboratory reagent water, such as HPLC grade. For soil samples the method blank is an aliquot of control soil, such as Ottawa sand, processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside of the control limits. Re-extraction of the blank, other batch QC, and the affected

samples are required when the method blank is deemed unacceptable. See QA Policy 003 for specific acceptance criteria.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix, such as Ottawa sand, and spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside of the control limits provided by the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See QA Policy 003 for specific acceptance criteria.

9.3. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. A MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside of the control limits must be within the control limits in the LCS. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte are outside of the control limits provided by the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field samples, and the MS/MSD may be required after evaluation and review.

9.4. A duplicate control sample (LCSD or DCS) must be substituted when insufficient sample volume is provided to process an MS/MSD pair. The LCSD is evaluated in the same manner as the LCS. See QA Policy 003 for specific acceptance criteria.

9.5. A second source calibration standard must be analyzed with each initial calibration curve. Each compound of the second source calibration standard must be within +/- 35% of its expected value.

10. CALIBRATION AND STANDARDIZATION

10.1. A fixed injection volume is used for quantitation purposes and is to be the same for both the sample and standards on the LC/MS.

10.2. Initial Calibration Curve - Five analytical standards of different analyte concentrations are used to generate the curve.

10.3. If a second order calibration curve is used then six analytical standards are analyzed. The Coefficient of Determination, (r^2) must then be greater than 0.990 for the curve to be considered in control.

10.4. The initial calibration curve is injected at the beginning of the batch to obtain the area counts for each analyte at each concentration, and the response factor is calculated using the following formula (on Micromass Masslynx data system):

FORMULA1 $$Rf = \frac{A_S \times C_{is}}{C_S \times A_{is}}$$

where C_s is the concentration of the standard in ng/mL, A_s is the area count of the standard, C_{is} is the concentration of the internal standard, and A_{is} is the area count of the internal standard.

10.4.1. The average response factor (Rfa) and the percent relative standard deviation (% RSD) is determined for each analyte. The initial calibration is valid for each analyte if the % RSD is equal to or less than 30%; linearity can be assumed and the average response factor can be used in place of a calibration curve.

10.4.2. For a second order calibration curve, the coefficient of determination (r^2) must be greater than or equal to 0.990 for the initial calibration to be valid.

10.4.3. If the % RSD is greater than 30 % (or the r^2 is less than 0.990), then the cause must be determined to bring the system back in control before samples are analyzed, and the system must be re-calibrated.

10.5. All units used in the calculations must be consistently uniform, such as concentration in ng/mL and response in area.

10.6. Samples are analyzed immediately after the curve, if the curve has met criteria. A second source calibration standard is injected before running samples to verify the validation of the curve. The concentration for each analyte is determined by using the response factors from the initial calibration curve. The value calculated is compared to the expected value and should meet the percent difference (%D) criteria for each compound of less than 35%.

Formula 2 $$\%D \quad \frac{|R_1 - R_2|}{R_1} \times 100\ \%$$

where R1 is the expected concentration, and R2 is the calculated concentration based on the initial calibration.

10.7. The continuing calibration verification standard (CCV) is the midpoint standard, and throughout the run it is the same. It is injected singly for every 10 samples or less,

and at the end of the batch. The concentration for each compound is compared to the expected value and should meet the %D criteria for the standards are valid.

10.8. The %D for the CCV must be ≤ 30% to be valid in the analytical batch.

11. EXTRACTION PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and the QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

The SPE method has been approved by the USACOE and is used in place of the salting-out method.

11.3. Solid Phase Extraction for Explosives and Specialty Chemicals in Aqueous Samples.

Below is an overview of the program to run the workstation. Whenever the method is modified, edit and update the method on a 3.5 inch disk.

Step1: Process 6 samples using the following procedure:

Step 2: Condition column with 10 mL of acetonitrile into SOLVENT WASTE

Step 3: Condition column with 10 mL of water into AQUEOUS WASTE

Step 4: Condition column with 5 mL of HOAc/water 0.1% into AQUEOUS WASTE

Step 5: Load 950 mL of sample onto column

Step 6: Rinse column with 5 mL of HOAc/Water 0.1% into AQUEOUS WASTE

Step 7: Dry Column with gas for 2.0 minutes

Step 8: Collect 5.0 mL fraction into sample tube using HOAc/MeCN 0.1%

Step 9: END

FLOW RATES	mL/min
Conditioning flow	15.0
Load flow	15.0
Rinse flow	5.0
Elute flow	2.0
Conditioning Air Push	15.0
Rinse Air Push	20.0
Elute Air Push	5.0
SPE PARAMETERS	
Push Delay	5 sec
Air Factor	1.0
Autowash Volume	1.00 mL
WORKSTATION PARAMETERS	
Maximum Elution Volume	12.0 mL
Exhaust Fan on	Y Y=Yes N=No
Beeper on	Y Y=Yes N=No
NAME SOLVENTS	
Solvent 1	water
Solvent 2	methanol
Solvent 3	acetonitrile
Solvent 4	HOAc/Water 0.1%
Solvent 5	HOAc/MeCN 0.1%

where HOAc/Water is acetic acid in water at 0.1% v/v, and HOAc/MeCN is acetic acid in acetonitrile at 0.1% v/v.

gas = compressed air or nitrogen.

WARNING: NEVER USE ACETONE ON THE **ZYMARK** AUTOTRACE SPE.

11.3.1. Setup of samples for extraction on the Zymark Autotrace Workstation, allow the samples to equilibrate to room temperature.

11.3.2. Measure 1000 mL of aqueous sample into amber glass bottles.

- For MB, DCS, and LCS, use nanopure water.

11.3.2.1. If samples contained excess particulates, either decant the sample very carefully or filter through Whatman fiber filter to obtain a clear solution then measure 1000 mL of aqueous sample into amber glass bottles.

11.3.3. Add the surrogates to all samples, including the MB, LCS and MS/SD or the DCS.

- Add 50 uL of the 50 ug/mL 8321-Surrogate (nitrobenzene-d5) standard to yield 2.5 ppb.

11.3.4. Fortify with the appropriate solutions to the DCS, LCS, and MS/MSD.

- Add 50 uL of the 50 ug/mL 8321 mix to yield 2.5 ppb.
- Add 100 uL of the 200 ug/mL NG/PETN mix to yield 20 ppb.

11.3.5. Mix the contents well.

11.3.6. Weigh and record the initial mass of each sample bottle, including the QC's (with the screw caps on).

11.3.7. The samples are ready for extraction by the Zymark Autotrace SPE workstation.

11.3.8. The program for the Autotrace SPE workstation must be set up using the PC computer on a 3.5" disk. The overview of the program and the parameters are shown at the beginning of the section.

11.3.9. Before starting the run, check the solvents and replace the solvents if necessary. If necessary, the solvent lines should be purged by loading the solvent purging program.

11.3.10. The sample lines should have been in a plastic bag or in a glass jar, indicating the lines have been cleaned with methanol followed by nanopure water using the Cleaning Sample Lines program.

11.3.11. Set the SPE columns in place and depress the plunger. Once in place the green light above the depressing leveler should be on.

11.3.11.1. Use 6-mL, 500 mg Porapak RDX Cartridges. Waters Catalog number WAT047220, or equivalent. Record the manufacturer and lot number.

11.3.12. Set the receiving vessels (or test tubes) in place. Be sure the first sample is on your left and the sixth sample on your right when facing the unit.

11.3.13. Check the level and identity of the solvents - replace if necessary, and set the samples in its place.

11.3.14. Check the waste containers (Aqueous and Organic) and replace if necessary. Do NOT allow waste containers to overflow.

11.3.15. Load the program '8321 EXP-A' in the disk drive of the Zymark Autotrace, and press Load.

11.3.16. When the disk has been loaded, the Zymark Autotrace is ready to start. Follow instructions displayed on the screen "Press CONT to start" when ready to start.

11.3.17. When the system is completed, the screen displays "program completed". Move the extracts in the test tubes aside and seal them with teflon-lined screw caps. Remove the SPE columns and discard them.

11.3.18. Measure and record the final mass of the sample bottles (with caps).

11.3.19. Clean sample lines with methanol follow by water. Insert the sample lines in a container containing methanol, and run the 'Cleaning Sample Lines program' using emptied columns (or rinse the plunger with methanol and depress the plunger without columns), and repeat the procedure with water.

11.3.20. After the lines are cleaned, continue with next set of samples or turn off the Zymark - place the sample lines in a plastic bag.

11.3.21. The acetonitrile extracts collected in 8-mL test tubes; adjust the final volume to 5.0 mL with 0.1% acetic acid in acetonitrile. Mix the contents well.

11.3.22. Filter the acetonitrile extract through an Acrodisc LC25 filter assembly using a 10-cc disposable syringe into a new test tube. DO NOT RE-ADJUST THE VOLUME.

11.3.23. The acetonitrile extracts (concentration = 950mL/5.0mL) are stored in the refrigerator at 4 ± 2°C . Dilutions are required for analysis.

11.3.24. Dilute the extracts 4X with water by aliquoting 1.0 mL of the extracts into a 4-cc vial, and add 3.0 mL of water. Mix the contents well.

11.3.25. Add internal standard (8321-EXP-IS) to a final concentration of 50 ng/mL: On a 4.0 mL extract add 20 uL of the 10 ug/mL 8321-EXP-IS mix. Mix the contents well.

11.3.26. The extracts are stored in the refrigerator at 4 ± 2°C until ready for analysis. The final extract concentration is 950mL/20mL.

11.4. Soil/Sediment Samples.

NOTE: Do NOT grind samples containing high concentrations of explosives. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.

11.4.1. Subsample approximately 15 to 30 grams of soil into a disposable weighing boat for drying. Other types of container may be used.

11.4.2. If the samples do not appear homogeneous and not suspicious, dry the samples in a cool ventilated area and away from direct light. **DO** NOT HEAT the samples.

11.4.2.1. Drying time of soil samples depend on how wet the samples are received. A typical time for a damp soil to dry is approximately 8 to 24 hours with the soil spread thin on the weighing boat.

11.4.3. If samples do appear suspicious, stop work and consult with the project manager and client regarding possible high levels of explosives.

11.4.4. After the samples are dried, grind thoroughly in a Waring blender (items previously rinsed with acetonitrile and dried).

11.4.5. Sift the sample through a sieve size 30 mesh. If samples are not to be extracted on the same day, store the sifted soil in the freezer and protect from light.

11.4.6. When the extractions are to be performed, turn on the re-circulating cooler to cool the sonication bath to operating temperature (9 to 30°C). The sonication bath generates heat when the power is on.

- If the cooling system fails during extraction, observe and record the bath temperature. Temperature near 45°C may cause degradation of the components. If applicable, analyze the LCS and MS/SD to determine if the batch are out of control. Temperature near 80°C has caused degradation and the batch must be re-extracted.

11.4.7. Weigh 2.0 grams of dried, homogeneous sample into 40-mL glass vials.

- For control, such as the MB and LCS or DCS, use Ottawa sand. If a larger sample size is desired, then the ratio of sample to solvent must be consistent to 1 gram/5 mL.

11.4.8. Add the surrogate to each sample, MB, and LCS and MS/MSD or DCS.

- Add 100 uL of the 50 ug/mL 8321-Surrogate (nitrobenzene-d5) standard to yield 2.5 ppm.

11.4.9. Fortify the LCS and MS/MSD or the DCS.

- Add 100 uL of the 50 ug/mL 8321 standard mix to yield 2.5 ppm.
- Add 100 uL of the 200 ug/mL NG/PETN mix to yield 20 ppm.

11.4.10. Add a total volume of 10.0 mL of 0.1% acetic acid in acetonitrile, sealed the vial with a screw cap, and shake briefly to mix the contents well.

11.4.10.1. To the samples containing the surrogate, add 9.9 mL of the extracting solvent.

11.4.10.2. To the samples containing the surrogate and 8321 mix, add 9.8 mL of the extracting solvent.

11.4.10.3. To the samples containing the surrogate and NG/PETN mix, add 9.7 mL of the extracting solvent.

11.4.10.4. To the samples containing the surrogate, 8321 mix and the NG/PETN mix, add 9.6mL of the extracting solvent.

11.4.11. Place the samples in the ultrasonic bath, and sonicate for 18 hours. Keep the water bath cooled at 35°C or cooler with the re-circulating cooler, and protect the samples from light by covering the sonication bath.

11.4.12. When the sonication is completed, allow the samples to settle for 30 minutes or longer, or centrifuge the samples at approximately 1,200 rpm for 10 minutes.

11.4.13. Decant or filter the extracts (minimally 4 mLs is needed) into 8-cc test tubes. The extracts may be worked up immediately or stored in the refrigerator at 4 ± 2°C until ready for further prep.

11.4.14. The acetonitrile extracts (concentration = 2.0g/10mL) are stored in the refrigerator at 4 ± 2°C. Dilutions are required for analysis.

11.4.15. Dilute the extracts 4X with water by aliquoting 1.0 mL of the extracts into a 4-cc vial, and add 3.0 mL of water. Mix the contents well.

11.4.16. Add internal standard (8321-EXP-IS) to a final concentration of 50 ng/mL: On a 4.0 mL extract add 20 uL of the 10 ug/mL 8321-EXP-IS mix. Mix the contents well.

11.4.17. If the extracts have not been filtered or are cloudy, filter the extracts through an Acrodisc LC25 filter assembly using a 10-cc disposable syringe into a new vial. DO NOT RE-ADJUST THE VOLUME.

11.4.18. The extracts are stored in the refrigerator at 4 ± 2°C until ready for analysis. The final extract concentration is 2.0g/40mL.

11.5. INSTRUMENTATION for Explosives and Specialty Chemicals by **LC/MS**

NOTE: The conditions listed below are the recommended analytical conditions for the analysis of the 8321 compounds. If changes to the LC/MS conditions are necessary, they will be noted on the chromatogram.

11.5.1. Primary Column: Carbosorb ODS-2, 250 x 4.6mm x 5 um, or equivalent.

11.5.2. Column Temperature: 35°C (heater box)

11.5.3. Mobile Phase for 8321 Explosives.

RESERVOIR	SOLVENT for 8321 EXP	Solvents for 8321 EXP+ NG/PETN
A	Water	Water with 10mM Ammonium Acetate
B	Acetonitrile	Acetonitrile
C	Methanol	Methanol with 10mM

	Ammonium Acetate

11.5.p. Gradient Program for LC/TSP/MS. The following conditions must be optimized whenever possible to provide the necessary separation.

Time minutes	Flow mL/min	%A	%B	%C	Curve
initial	0.8	35	5	60	--
12	0.8	35	5	60	1
12.5	0.8	0	100	0	6
15	0.8	35	5	60	11

11.5.5. Acquisition time is 15 minutes with approximately 10 minutes for the LC system to equilibrate before the next sample.

11.5.6. Injection volume: a fixed amount such as 100 uL, depending on the sensitivity of the10 ng/mL standard.

11.5.7. Instrument conditions - The following conditions must be optimized whenever possible to provide good performance.

Parameters	Setting	Range
Ion mode	APCI -	APCI -/APCI +/ EI -/ EI +
Corona voltage	35 uA	0 – 35
Cone voltage	1-25 v[1]	0 – 350
Extractor	2 v	0 – 410
Rf lens	0.6 v	0 – 1.0
Cone Gas flow	100 L/hr	0 – 300 MAX
Desolvation Gas Flow	400 L/hr	0 – 800 MAX
Source Temperature	100 °C	0 – 150
Desolvation Temperature	500 °C	0 – 600 +
Multiplier	780 v	0 – 1000 +

11.5.8. Conditions below describe analysis in negative-ion multiple reaction monitoring (MRM) for 8321 Explosives. Ions used in other type of monitoring will differ from those listed below and must be established by analysis of standard reference materials.

Analytes	Quantifying Trace	Retention Time	Cone Voltage

8321 Components[1]-			
HMX	102 > 102	3.90	18
RDX	102 > 102	4.86	18
IS_1; RDX-$^{13}C_3$	104 > 104	4.83	18
1,3,5-Trinitrobenzene	213 > 213	6.05	25
TETRYL	241 > 214	6.45	25
IS_2; 1,3-DNB-d_4	172 > 172	6.74	25
1,3-Dinitrobenzene	168 > 168	6.78	25
Surr; NB-d_5	128 > 128	7.33	18
Nitrobenzene	123 > 123	7.42	18
4-Amino-2,6-dinitrotoluene	197 > 197	7.34	21
2-Amino-2,4-dinitrotoluene	197 > 197	7.72	21
2.4.6-Trinitrotoluene	227 > 227	7.73	22
IS_3; 2,6-DNT-d_3	185> 185	8.33	20
2.6-Dinitrotoluene	182> 182	8.38	10
2,4-Dinitrotoluene	182 > 182	8.71	10
2-Nitrotoluene	137 > 137	9.77	13
4-Nitrotoluene	137 > 137	10.31	13
3-Nitrotoluene	137 > 137	10.88	13
Specialty Chemicals-			
Nitroglycerin	NA	NA	NA
PETN	NA	NA	NA
Air Train Standards-			
2,4-Dinitroflurobenzene	185 > 185	6.32	10
TNT-$^{13}C_6$-$^{15}N_3$	237 > 237	7.73	10

1 = Compounds listed in approximate retention time order.

NA = Not Applicable

Quantifying trace – The chemical ionization technique is a soft or low energy ionzation process resulting in a molecular ion for each analyte with little or no fragmentation. The collision energy is set at 1.0.

Cone voltage is determined when optimizing instrument response for each analyte. When fragmentations are desired to produce daughter ion(s), the collision energy is also determined for each analyte when optimizing the instrument conditions.

11.5.9. Conditions below describe analysis in negative-ion selected ion recording (SIR) mode for 8321 Explosives including NG and PETN.

Analytes	Quantifying	Retention	Cone

	Trace	Time	Voltage
18321 **Components**[1]-			
HMX	102	4.03	18
RDX	102	4.96	18
IS_1; X-$^{13}C_3$	104	4.96	18
1,3,5-Trinitrobenzene	213	6.23	25
TETRYL	241	6.62	25
IS_2; 1,3-DNB-d_4	172	6.91	25
1,3-Dinitrobenzene	168	6.96	25
Surr; NB-d_5	128	7.50	18
Nitrobenzene	123	7.64	18
4-Amino-2,6-dinitrotoluene	197	7.55	21
2-Amino-2,4-dinitrotoluene	197	7.94	21
2,4,6-Trinitrotoluene	227	7.99	22
IS_3; 2,6-DNT-d_3	185	8.57	20
2,6-Dinitrotoluene	182	8.62	10
2,4-Dinitrotoluene	182	8.96	10
2-Nitrotoluene	137	10.14	13
4-Nitrotoluene	137	10.67	13
3-Nitrotoluene	137	11.25	13
Specialty Chemicals-			
Nitroglycerin	289	7.25	8
PETN	240	10.18	5

1 = Compounds listed in approximate retention time order.

11.5.10. Change in-line-frit as needed to retain proper pressure, peak shape and response.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Results are reported in mg/Kg (ppm) for soil and ug/L (ppb) for aqueous, unless otherwise instructed.

12.2. Results are reported as dry weight since soil samples are air dried, ground with mortar and pestle (or with a blender), and sieved, unless otherwise specified by the client.

12.3. The concentration of each analyte and surrogate in a sample is calculated by using the following formula:

FORMULA Amount Found (ppb) = $(RF_a \times A_x \times V_x \times C_{is}) / (W_s \times A_{is})$

12.4. The results of soils in ppb (ug/Kg) are converted to ppm (mg/Kg) by dividing the result in ug/Kg by 1000; mg/Kg = (X ug/Kg) / 1000 ug/mg

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure this procedure is to be performed by an associate who has been properly trained in its use and has the required expertise.

14. POLLUTION PREVENTION

14.1. No solvents of any kind or in any amount are to be disposed of in the sinks or evaporated in the hoods.

15. WASTE MANAGEMENT

15.1. Waste management in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator or Hazardous Materials Technician should be contacted if additional information is required.

15.2. All waste must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS002, Table 1.

15.3. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS003, Disposal of Samples After Analysis.

16. REFERENCES

16.1. SW-846 Draft Method 8330, "Nitroaromatics and Nitramines by High Pressure Liquid Chromatography (HPLC)", Revision 0, November 1992, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

16.2. Thomas F. Jenkins, et. al., "Comparison of Cartridge and Membrane Solid-Phase Extraction with Salting-Out Solvent Extraction for Preconcentration of Nitroaromatic and Nitramine Explosives from Water", Special Report 92-95, December 1992, US Army Corps of Engineers, Cold Regions Research & Engineering Laboratory, Hanover, New Hampshire.

16.3. "Quanterra Quality Control Program", QA-003, Quanterra Environmental Services, Revision No. 0, April 4, 1995.

16.4. "Laboratory Documentation Practices", LP-CAL-0001, Quanterra Environmental Services, Revision 3.0, December 5, 1991.

16.5. "Calibration and Calibration Check of Balances", SAC-QA-0041, Quanterra Environmental Services, Revision 1.1, December 30, 1994.

17. MISCELLANEOUS

17.1. Estimated detection limit (EDL) for 8321-EXP/SPEX in aqueous and soil.

Analytes	Aqueous, ppb	Soil, ppm
8321 Mix	0.21	0.20
NG	5.0	4.0
PETN	2.5	2.0

17.2. Fortification levels for 8321-EXP/SPEX in aqueous and soil.

Analytes	Aqueous, ppb	Soil, ppm
NB-d_5 (surr)	2.5	2.5
NG	20	20
PETN	20	20
8321 Mix	1.0	2.0

17.3. MDL spike level for 8321-EXP/SPEX in aqueous and soil.

Analytes	Aqueous, ppb	Soil, ppm
8321 Mix	0.10	0.10
NG	2.5	2
PETN	2.5	2.0

17.4. Deviations from reference method.

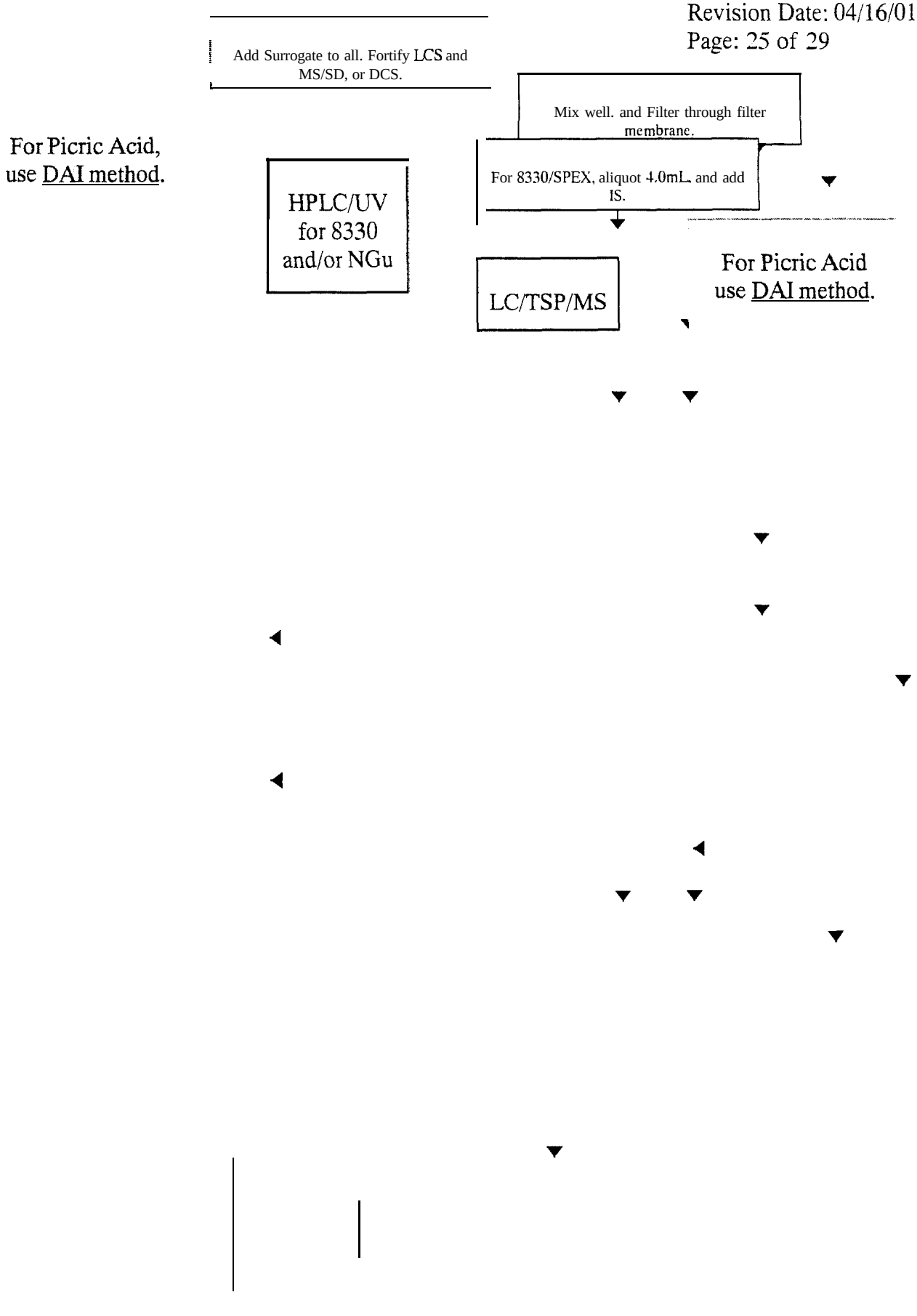
17.4.1. This method deviates from EPA method 8330 extraction method, November 1992 revision on the following:

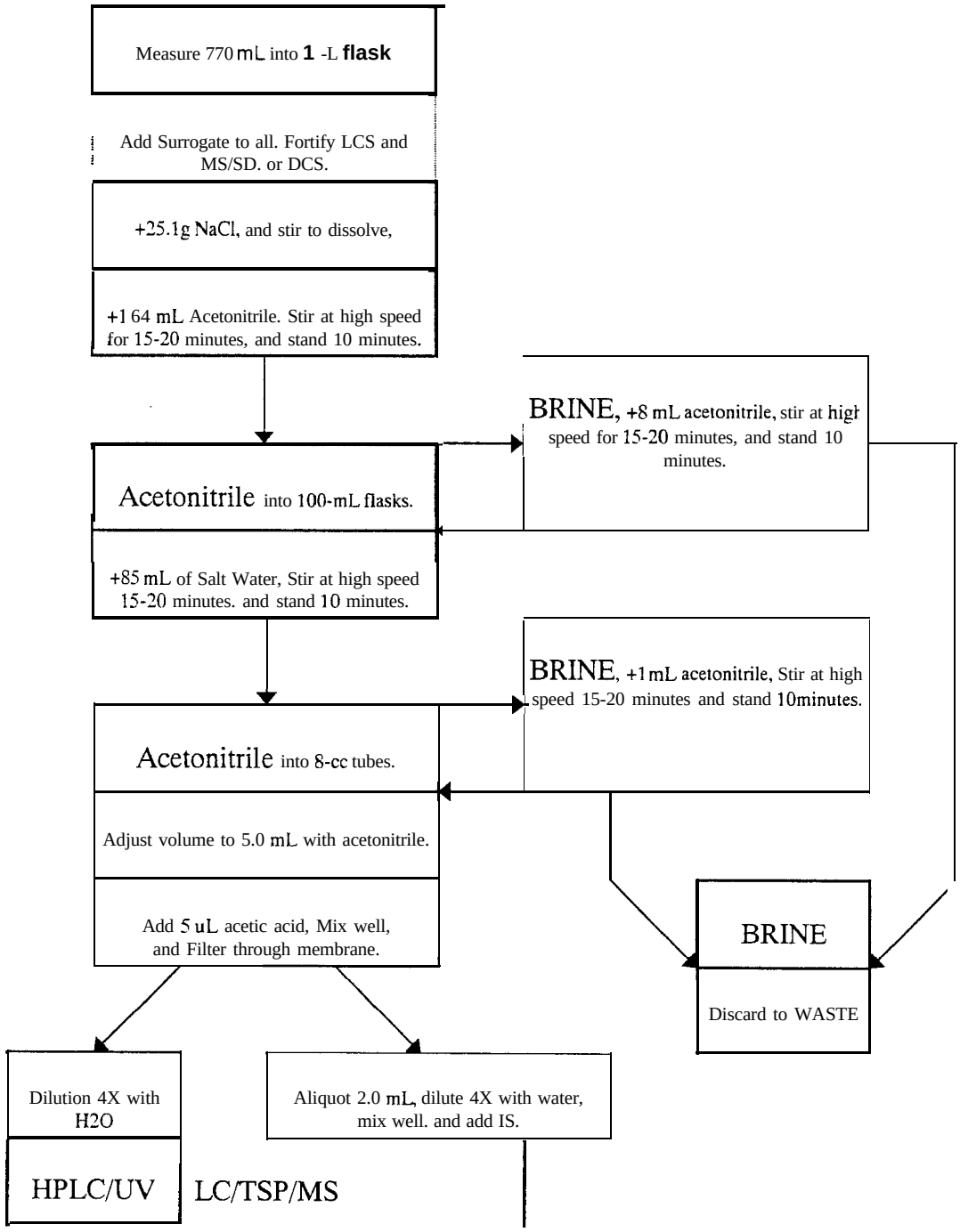
Acetonitrile is our preferred solvent instead of methanol in the preparation of standards.

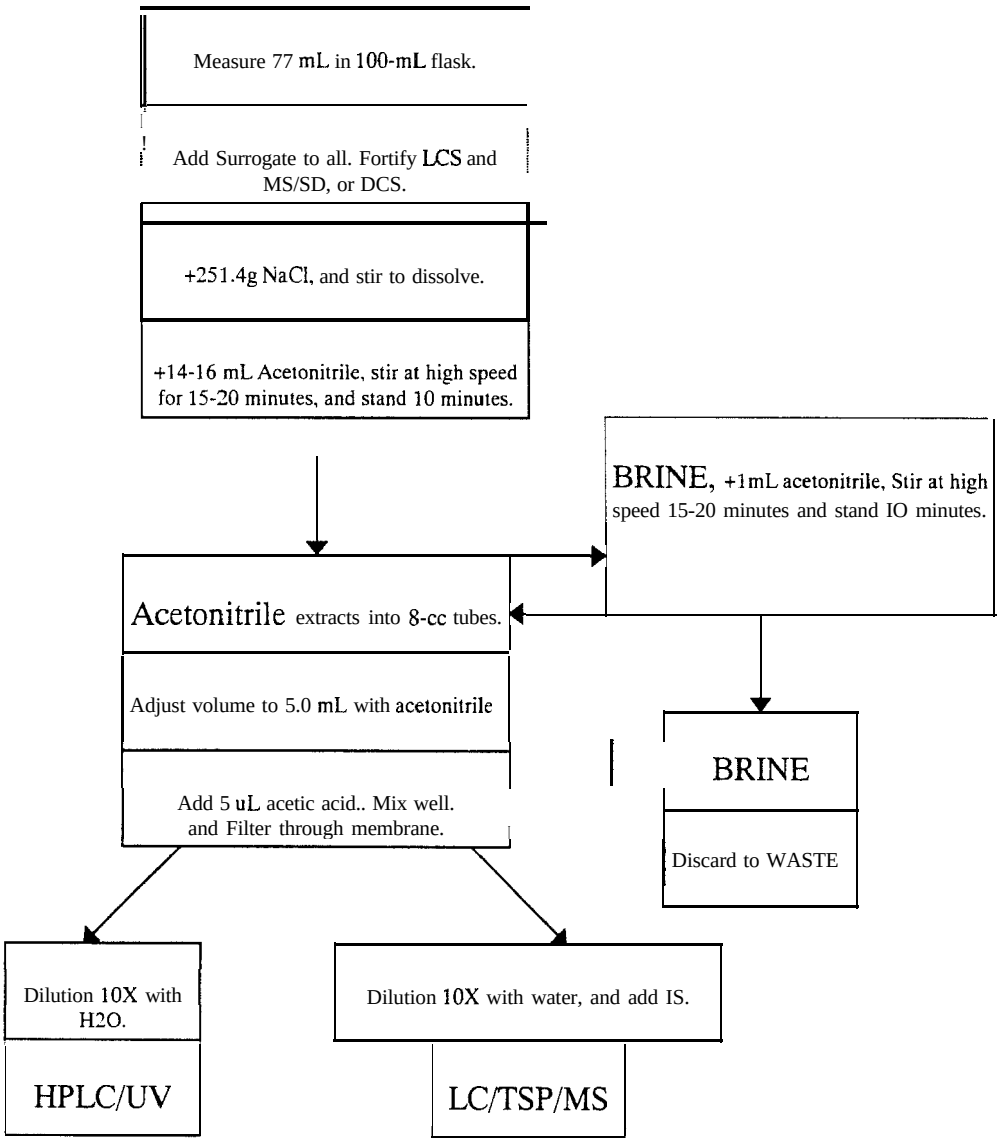
The final solvent for extracts for HPLC/UV analysis should be no more than 25% acetonitrile in water. Higher percentages of acetonitrile causes poor chromatography.

The initial calibration is performed by injecting five concentration levels singly instead of triplicate injections of five levels randomly.

Calcium chloride solution is a flocculating agent that aids in the filtration of soil extracts. The solution is not used if the samples are to be performed on the LCMS as it may cause clogging of the APCI unit, in-line frits, and over-pressurizing the HPLC.


Add Surrogate to all. Fortify LCS and MS/SD, or DCS.
Mix well. and Filter through filter membranc.
For 8330/SPEX, aliquot 4.0mL and add IS.
For Picric Acid, use DAI method.
HPLC/UV for 8330 and/or NGu
LC/TSP/MS
For Picric Acid use DAI method.


Measure 770 mL into 1 -L flask
Add Surrogate to all. Fortify LCS and MS/SD. or DCS.
+25.1g NaCl, and stir to dissolve,
+164 mL Acetonitrile. Stir at high speed for 15-20 minutes, and stand 10 minutes.
BRINE, +8 mL acetonitrile, stir at high speed for 15-20 minutes, and stand 10 minutes.
Acetonitrile into 100-mL flasks.
+85 mL of Salt Water, Stir at high speed 15-20 minutes. and stand 10 minutes.
BRINE, +1 mL acetonitrile, Stir at high speed 15-20 minutes and stand 10minutes.
Acetonitrile into 8-cc tubes.
Adjust volume to 5.0 mL with acetonitrile.
Add 5 uL acetic acid, Mix well, and Filter through membrane.
BRINE
Discard to WASTE
Dilution 4X with H2O
Aliquot 2.0 mL, dilute 4X with water, mix well. and add IS.
HPLC/UV
LC/TSP/MS


Measure 77 mL in 100-mL flask.
Add Surrogate to all. Fortify LCS and MS/SD, or DCS.
+251.4g NaCl, and stir to dissolve.
+14-16 mL Acetonitrile, stir at high speed for 15-20 minutes, and stand 10 minutes.
BRINE, +1mL acetonitrile, Stir at high speed 15-20 minutes and stand IO minutes.
Acetonitrile extracts into 8-cc tubes.
Adjust volume to 5.0 mL with acetonitrile
Add 5 uL acetic acid.. Mix well. and Filter through membrane.
BRINE
Discard to WASTE
Dilution 10X with H2O.
HPLC/UV
Dilution 10X with water, and add IS.
LC/TSP/MS

STL Sacramento

STANDARD OPERATING PROCEDURE CHANGE FORM

SOP Title:	
SOP Sections Affected by Change:	
Reason for Addition or Change:	
Change Effective From [Date]:	
Change or Addition (Specific Section; use additionai sheets if necessary.):	

Submitted by/Date:

Approved by: *

Technical Reviewer: *Date:*

EH&S Signature: *Date:*

QA Signature: *Date:*

Management Signature: *Date*

Must be same signature authorities of SOP being revised.

Compound Name	Replicate Measurement (ug/L)							Calculations			MDL Spike Conc	Barksdale, WI RL	MDL > 10% Spike Conc	MDL < LCL	MDL < spike conc
	MDL #1	MDL #2	MDL #3	MDL #4	MDL #5	MDL #6	MDL #7	Ave	STDEV	MDL					
HMX	0.0917	0.1102	0.0914	0.1226	0.1025	0.0779	0.1169	0.1019	0.0159	0.0499	0.10	0.25		yes	yes	yes
RDX	0.0911	0.1064	0.0976	0.1010	0.0939	0.0972	0.1095	0.0995	0.0066	0.0207	0.10	0.25		yes	yes	yes
1,3,5-Trinitrobenzene	0.0973	0.0858	0.0966	0.1038	0.0975	0.0944	0.0940	0.0956	0.0054	0.0170	0.10	0.25		yes	yes	yes
ETRYL TERRYL	0.1004	0.1097	0.1177	0.1156	0.1099	0.1123	0.1170	0.1118	0.0060	0.0188	0.10	0.50		yes	yes	yes
1,3-Dinitrobenzene	0.0957	0.0963	0.1068	0.0968	0.0969	0.1096	0.0965	0.0998	0.0058	0.0183	0.10	0.10		yes	yes	yes
(Surr) d5-Nitrobenzene	2.5774	2.4787	2.6391	2.4925	2.3512	2.5549	2.4936	2.5125	0.0912	0.2868	2.50	NA		NA	NA	NA
Nitrobenzene	0.0763	0.0851	0.0840	0.0809	0.0884	0.0810	0.0873	0.0833	0.0042	0.0132	0.10	0.25		yes	yes	yes
4-Amino-2,6-DNT	0.0987	0.0926	0.0926	0.0901	0.0997	0.1013	0.0877	0.0947	0.0052	0.0164	0.10	0.10		yes	yes	yes
2-Amino-4,6-DNT	0.0851	0.0806	0.0884	0.0843	0.1060	0.0866	0.0811	0.0874	0.0086	0.0272	0.10	0.10		yes	yes	yes
2,4,6-Trinitrotoluene	0.0877	0.0948	0.0870	0.0918	0.0850	0.0922	0.0912	0.0900	0.0035	0.0109	0.10	0.10		yes	yes	yes
2,6-Dinitrotoluene	0.0938	0.0867	0.0855	0.0957	0.0834	0.0928	0.0856	0.0891	0.0049	0.0153	0.10	0.005		yes	NO*	yes
2,4-Dinitrotoluene	0.0842	0.0925	0.0858	0.0749	0.0776	0.0896	0.0827	0.0839	0.0062	0.0196	0.10	0.005		yes	NO*	yes
2-Nitrotoluene	0.0711	0.0802	0.0745	0.0747	0.0885	0.0806	0.0791	0.0784	0.0057	0.0178	0.10	0.25		yes	yes	yes
4-Nitrotoluene	0.0869	0.0838	0.0736	0.0815	0.0930	0.0974	0.0861	0.0860	0.0077	0.0243	0.10	0.25		yes	yes	yes
3-Nitrotoluene	0.0870	0.0815	0.0797	0.0844	0.0823	0.1004	0.0768	0.0846	0.0077	0.0242	0.10	0.25		yes	yes	yes

Sample Size: 1000 mL Test: 8321 E instrument: A3A MDL(ug/L) = STDEV * 3.143

Date of MDL: 4/11/01 Matrix: water Column: Carbosorb-ODS

Analyst: DEG/SDR Spike Amt: 0.1-2.5 PPB

* Lab MDL's are above 0.005 ug/L: lab will report to 0.05 ug/L

Compound Name	Replicate Measurement (ng/ml)							Calculations			MDL	Barksdale, WI	MDL > 10% Spike Conc	MDL < LCL	MDL < spike conc
	MDL #1	MDL #2	MDL #3	MDL #4	MDL #5	MDL #6	MDL #7	Ave	STDEV	MDL	Spike Conc	RL			
Nitroglycerin	3.2552	3.3382	3.3443	3.2303	3.3320	3.4541	3.1905	3.3064	0.0883	0.2775	2.50	2.50		yes	yes	yes
PETN	3.0405	2.5647	2.4344	2.7165	2.3750	2.6773	3.1260	2.7336	0.2930	0.9210	2.50	10.00		yes	yes	yes
2,5-Nitrobenzene	2.4641	2.5963	2.3950	2.2709	2.4709	2.9700	2.2239	2.4887	0.2511	0.7892	2.50	NA		NA	NA	NA

Sample Size: 1000 mL Test: 8321 E Instrument: A3A MDL(ng/ml) = STDEV * 3.143

Date of MDL: 4/17/2001-4/18/01 Matrix: water Column: Carbosorb-ODS

Analyst: DEG/JRB Spike Amt: 2.5 PPB

STL Sacramento

Method Proficiency Demonstration (DOC)

Date Completed:	11-Apr-01	Prep Analyst	Dennis Gall
Method ID	8321	Analytical Analyst	Dennis Gall
Method Description	8321 Explosives-A ug/L		
Prep Method	SPE		

		Amount Recovered				Percent Recovery							Control Limits		
Analyte	Spike ug/L	LCS 1 4/11/01 17:39	LCS 2 4/11/01 18:05	LCS 3 4/11/01 18:32	LCS 4 4/11/01 18:58	LCS 1 4/11/01 17:39	LCS 2 4/11/01 18:05	LCS 3 4/11/01 18:32	LCS 4 4/11/01 18:58	AVE %	AVE. ug/L	RSD	Lower	Upper	RSD
HMX	1.0	1.0293	0.9803	0.8846	1.0838	103%	98%	88%	108%	**99%**	**0.99**	8.5%			
RDX	1.0	0.9555	0.9692	0.9710	1.0126	96%	97%	97%	101%	**98%**	**0.98**	2.5%			
1,3,5-Trinitrobenzene	1.0	0.9925	0.8709	1.0391	0.9479	99%	87%	104%	95%	**96%**	**0.96**	7.4%			
Tetryl ~~TERRYL~~	1.0	1.0235	0.9168	0.8870	0.8469	102%	92%	89%	85%	**92%**	**0.92**	8.2%			
1,3-Dinitrobenzene	1.0	0.9897	0.9510	0.9903	0.9524	99%	95%	99%	95%	**97%**	**0.97**	2.3%			
d5-Nitrobenzene	2.5	2.7892	2.4439	2.6963	2.5142	112%	98%	108%	101%	104%	2.61	6.1%			
Nitrobenzene	1.0	1.0029	0.8706	0.9834	0.9142	100%	87%	98%	91%	**94%**	**0.94**	6.5%			
4-Amino-2,6-DNT	1.0	1.0393	0.9665	0.9669	0.9281	104%	97%	97%	93%	98%	**0.98**	4.8%			
2-Amino-4,6-DNT	1.0	0.9766	0.9590	0.8987	0.8445	98%	96%	90%	84%	**92%**	**0.92**	6.5%			
2,4,6-Trinitrotoluene	1.0	0.9580	0.9753	0.9549	0.8839	96%	98%	95%	88%	**94%**	**0.94**	4.3%			
2,6-Dinitrotoluene	1.0	0.9901	0.9631	0.9536	0.8741	99%	96%	95%	87%	**95%**	**0.95**	5.3%			
2,4-Dinitrotoluene	1.0	1.0261	0.9807	0.9343	0.9629	103%	98%	93%	96%	**98%**	**0.98**	3.9%			
2-Nitrotoluene	1.0	1.0420	0.9656	0.9232	0.9106	104%	97%	92%	91%	**96%**	**0.96**	6.2%			
4-Nitrotoluene	1.0	0.9613	0.9536	0.9054	0.8876	96%	95%	91%	89%	**93%**	**0.93**	3.9%			
3-Nitrotoluene	1.0	0.93771	0.98631	0.90771	0.8712	94%	99%	91%	87%	**93%**	**0.93**	5.3%			
Internal Standards															
13C3-RDX	50.0					0%	0%	0%	0%	#DIV/0!	#DIV/0!	#DIV/0!			
1,3-Dinitrobenzene-d4	50.0					0%	0%	0%	0%	#DIV/0!	#DIV/0!	#DIV/0!			
2,6-Dinitrotoluene-d3	50.0					0%	0%	0%	0%	#DIV/0!	#DIV/0!	#DIV/0!			

Note: Both averages are calculated based on the amount recovered, not the calculated recovery.
RSD's are calculated based on the amount recovered, not the calculated recovery.

STL Sacramento

Method Proficiency Demonstration (DOC)

Date Completed:	17-Apr-01	Prep Analyst	Dennis Gall
Method ID	8321	Analytical Analyst	Dennis Gall
Method Description	8321 Explosives-A ug/L		
'rep Method	SPE		

		Amount Recovered				Percent Recovery							Control Limits		
rnalyte	Spike ug/L	LCS 1 4/17/01 19:01	LCS 2 4/17/01 19:26	LCS 3 4/17/01 19:52	LCS 4 4/17/01 20:17	LCS 1 4/17/01 19:01	LCS 2 4/17/01 19:26	LCS 3 4/17/01 19:52	LCS 4 4/17/01 20:17	AVE %	AVE. ug/L	RSD	Lower	Upper	RSD
Nitroglycerin	25.0	24.4181	26.2461	27.8878	27.2328	98%	105%	112%	109%	106%	26.45	5.7%			
PETN	25.0	24.3242	30.9885	30.6111	31.8496	97%	124%	122%	127%	118%	29.44	11.7%			
d5-Nitrobenzene	2.5	2.2828	2.5846	2.6496	2.5019	91%	103%	106%	100%	100%	2.50	6.4%			
ıternal Standards															
1,3-Dinitrobenzene-d4	50.C					0%	0%	0%	0%	#DIV/0!	#DIV/0!	#DIV/0!			
2,6-Dinitrotoluene-d3	50.C					0%	0%	0%	0%	#DIV/0!	#DIV/0!	#DIV/0!			

Note: Both averages are calculated based on the amount recovered, not the calculated recovery.
RSD's are calculated based on the amount recovered, not the calculated recovery.

Standard Operating Procedure
Analytical Method

TITLE: Determination of Nitroglycerin in Soil/Sediment Samples by High Performance Liquid Chromatography (HPLC)

DEPARTMENT: Semivolatile Organics

APPLICATION:

This method covers the determination of the following explosive compound, Nitroglycerin (CAS# 55-63-0), in soils and sediments using High-Performance Liquid Chromatography (HPLC). The method detection limit for this compound, which represents the target detection limit that can be achieved in soil/sediment matrices using this method, and the Estimated Quantitation Limit (EQL) are presented in Attachment 1.

REFERENCE: Test Methods for Evaluating Solid Wastes
SW-846 Method 8330 (Revision 0, September 1994)

PROCEDURE SUMMARY:

Homogenized portions of soil/sediment samples are extracted with acetonitrile in a chilled ultrasonic water bath. The resulting extract is then taken through a 0.2 µm PTFE syringe filter ("acrodisc"), and analyzed on a HPLC using UV for detection and quantitation of this compound.

REVIEWED BY: Daniel M. Rude DATE: 8-19-97
Daniel M. Rude
Group Leader
Organic Laboratory

REVIEWED BY: Amy L. Austin DATE: 8/19/97
Amy L. Austin
Quality Assurance Officer

APPROVED BY: Eric L. Thomas DATE: 8/20/97
Eric L. Thomas
Laboratory Manager

SAFETY PRECAUTIONS:

- The toxicity and carcinogenicity of the chemicals used in this method have not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each analyst is responsible for maintaining awareness of Occupational Safety and Health Administration regulations regarding the safe handling of the chemicals used in this method.

- Standard precautionary measures used for handling other organic compounds should be sufficient for the safe handling of the analyte targeted this method. The only extra caution that should be taken is when handling the analytical neat material for the explosives themselves and in rare cases where soil or waste samples are highly contaminated with the explosives.

- Observe all standard laboratory safety procedures as outlined in the En Chem, Inc. Safety Training Manual.

INTERFERENCES:

- Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample-processing hardware that may lead to discrete artifacts and/or elevated baselines in liquid chromatography. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

- Matrix interferences may be caused by contaminants that are co-extracted from the sample.. The extent of matrix interferences will vary considerably from source to source, depending on the nature and diversity of the matrix involved. The cleanup procedure provided in this method may be used to minimize or overcome such interferences.

APPARATUS:

- Volumetric flasks, 10 mL, 25 mL
- Volumetric syringes, 10 µL, 25 µL, 50 µL, 100 µL, 500 µL, and 1000 µL
- Disposable glass pipets
- Beakers, assorted sizes, Griffin
- 40-mL glass vials, screw top sterile VOA vials with Teflon®-lined screw caps
- 5-mL glass test tube with Teflon-lined screw caps
- Teflon or stainless steel spatulas
- 2-mL Luer Lock syringes
- Vials, glass, 2-mL capacity with crimp top for LC autosampler
- Vials, glass, 2-mL capacity with Teflon-lined screw cap for storage of stock standard, surrogate, and stock matrix spike solutions
- Balance, Sartorius model L310
- Ultrasonic Bath, Fisher Scientific model FS28
- Endocal Refrigerated Circulations Bath
- PTFE syringe filters, 0.2 µm, 25 mm diam., Gelman Sciences, Part No. 4521
- Analytical system complete with High-Performance Liquid Chromatograph and all required accessories including analytical column, gases, W detector, with spectral capabilities and recording integrator (a data system is required for measuring peak areas or peak heights and recording retention times).
- HPLC System Consisting of:
  Gradient pumping system, constant flow, Perkin-Elmer Corporation, Series 250
  Alltech BDS Cl8 column, 5-µ particle size diameter, 250 mm x 4.6 i.d., stainless steel column, Alltech No. 11145
  Autoinjector, ISS-100, Perkin-Elmer
  PE LC-101 Oven Column heater
  W absorbance detector, PE LC235C Photo Diode Array Detector

NOTE: Equivalent equipment may be substituted.

REAGENTS:

- Acetonitrile, HPLC grade
- Methanol, HPLC grade
- Calcium Chloride, reagent grade, J.T. Baker, Catalog No. 1311-01

- Milli-Q® Water (reagent water)
- Calcium Chloride/Water Solution - dilute 5 g of calcium chloride diluted to 1-L volume with Milli-Q water
- 3,4-Dinitrotoluene, 1.0 mg/mL in MeOH, AccuStandard or equivalent
- Nitroglycerin, 1.0 mg/mL in MeOH:ACN (1:1), AccuStandard or equivalent
- Ottawa sand, Fisher, purified by heating at 400°C for 4 hours in a shallow tray

NOTE: Equivalent reagents may be substituted.

STANDARD & SPIKING SOLUTION PREPARATION:

Preparation of Nitroslvcerin Standard Stock Solution

Prepare a 100 µg/mL nitroglycerin standard stock solution by diluting 1.0 mL of a 1.0 mg/mL commercially available nitroglycerin stock ampule. Bring to 10 mL final volume in a 10-mL volumetric flask with HPLC grade acetonitrile.

Preparation of Nitroslvcerin Calibration Standards

1. Calibration standards are prepared at five concentration levels from the nitroglycerin stock solution and the surrogate ampule solution. Immediately prior to analysis, 500 µL of each of the five calibration standards are mixed with 500 µL of the calcium chloride/water solution in a 2-mL amber GC crimp top vial. The solutions are mixed thoroughly and are now ready for injection. The concentrations listed below reflect the effective concentration prior to diluting each standard with the calcium chloride/water solution.

   1.1 To prepare Calibration Standard 60 us/L, dilute 15 µL of the 100 µg/mL nitroglycerin calibration stock solution and 10 µL of 1.0 mg/mL 3,4-dinitrotoluene (surrogate @ 400 µg/L) ampule, volumetrically to mark in 25-mL of acetonitrile.

   1.2 To prepare Calibration Standard 100 µg/L, dilute 25 µL of the 100 µg/mL nitroglycerin calibration stock solution and 15 µL of 1.0 mg/mL 3,4-dinitrotoluene (surrogate @ 600 µg/L) ampule volumetrically to mark in 25-mL of acetonitrile.

1.3 To prepare Calibration Standard 200 µg/L, dilute 50 µL of the 100 µg/mL nitroglycerin calibration stock solution and 25 µL of 1.0 mg/mL 3,4-dinitrotoluene (surrogate @ 1000 µg/L) ampule volumetrically to mark in 25-mL of acetonitrile.

1.4 To prepare Calibration Standard 300 µg/L, dilute 75 µL of the 100 µg/mL nitroglycerin calibration stock solution and 35 µL of 1.0 mg/mL 3,4-dinitrotoluene (surrogate @ 1400 µg/L) ampule, volumetrically to mark in 25-mL of acetonitrile.

1.5 To prepare Calibration Standard 400 µg/L, dilute 100 µL of the 100 µg/mL nitroglycerin calibration stock solution and 40 µL of 1.0 mg/mL 3,4-dinitrotoluene (surrogate @ 1600 µg/L) ampule volumetrically to mark in 25-mL of acetonitrile.

2. Just prior to analysis, 500-µL of each of the five working calibration standards are mixed with 500-µL of the calcium chloride/water solution in a 2-mL GC crimp top vial. The solutions are mixed thoroughly and are now ready for injection.

NOTE: All calibration standards (prepared by 1:1 dilution with calcium chloride/water solution) must be prepared fresh daily.

Preparation of Nitroslvcerin Spiking Solution

Dilute 1.0 mL of 1.0 mg/mL of commercially prepared nitroglycerin volumetrically to mark in 10 mL of methanol.

PROCEDURE:

Sample Homosenization

1. Dry soil samples in air at room temperature or colder to a constant weight. Do this by placing approximately 20 to 30 grams of a well mixed aliquot of wet sample to an aluminum weighing pan. Place the weighing pans with the sample under the hood and allow to air dry at room temperature or colder. Do not to expose the samples to direct sunlight.

2. Grind and homogenize the dried samples thoroughly.

Sample Extraction

1. Weigh 2.0 g of sand into a 40-mL VOA vial; this will represent the method blank.
2. Weigh 2.0 g of the sand into a second 40-mL VOA vial; this will be used for the control spike.
3. Weigh 2.0 g of each sample and MS/MSD into a 40-mL VOA vial.
4. Spike 10 µL of the 1.0 mg/mL 3,4-dinitrotoluene (surrogate) solution into each 40-mL vial.
5. Spike 30 µL of the 100 µg/mL nitroglycerin spiking solution into the control spike, matrix spike and matrix spike duplicate.
6. Add 10 mL of HPLC grade acetonitrile to each 40-mL vial.
7. Cap each of the 40-mL VOA vials and manually shake each vial for 2 minutes.
8. Place vials in a chilled ultrasonic bath and sonicate for 18 hours.
9. Remove vials from ultrasonic bath and allow sample particulate to settle and equilibrate to room temperature for at least 20 minutes.
10. Filter 2 mL of the supernatant extract through a 0.2 µm PTFE syringe filter (acrodisc) using a 2-mL Luer Lock syringe into an 5-mL culture tube.
11. Combine 500 µL of the filtered extract with 500 µL of 5 g/L calcium chloride water directly in an injection vial. Store the unused portion of the filtered extract in the refrigerator.
12. The sample extract is ready for HPLC analysis.

Recommended HPLC Materials and Operatins Conditions

ANALYTICAL COLUMN: Alltech BDS Cl8 column, 5-p particle size diameter, 250 mm x 4.6 mm i.d., stainless steel column, Alltech No. 11145

GUARD COLUMN: 20 mm C-18 packing

PUMP PARAMETERS:

Flow rate: 1.5 mL/min., Gradient with ACN and Milli-Q® water:

ACN:Water

Step 0. 35:65 for 5 minutes equilibration (curve 0)
Step 1. 80:20 for 20 minutes (curve 1)
Step 2. 100:0 for 3 minutes (curve 0)
Step 3. 35:65 for 1 minutes (curve 1)

Total run time, less equilibration time: 24 minutes

AUTO SAMPLER:

Sample loop: 150 µL
Volume injected: 100 µL

DETECTOR SETTINGS:

UV wavelength: 210 nm
Spectral Threshold = Trace

GAS: Helium 99.99+ percent purity for sparging mobile phase.

QUALITY CONTROL:

The minimum quality assurance/quality control operations necessary to satisfy the analytical requirements associated with the determination of the explosive compound listed in this method are as follows:

1. Method Blank Analysis

1.1 A laboratory method blank is a sample of reagent sand that is carried through the entire analytical sequence (extraction, dilution, and analysis).

1.2 A method blank is analyzed with every 20 samples processed or whenever samples are extracted, whichever is most frequent. It is the analyst's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, and glassware are minimized. An acceptable laboratory method blank should contain less than the reporting limit of any single explosive target compound.

1.3 If a laboratory method blank exceeds the above criteria, the analyst must consider the analytical system to be out of control. The source of the contamination must be investigated, and appropriate corrective action must be taken, namely re-extraction of all associated samples with the contaminated method blank.

2. Control Spike Analysis

2.1 The control spike is a reagent sand sample which is fortified with the matrix spike solution. The control spike may be from the same source as the blank or from an independent source. All compounds of interest will be spiked.

2.2 A laboratory control spike is prepared and analyzed with every 20 samples processed or whenever samples are extracted, whichever is most frequent.

2.3 Recoveries of the laboratory control spike are calculated using the equation in Step 3.2.

2.4 The control spike recovery must be evaluated by determining whether the concentration (measured as percent recovery) falls inside the advisory limits of 80% to 120%.

3. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

3.1 In order to evaluate the matrix effect of a sample on the analytical method and to provide both precision and accuracy information, an-MS/MSD analysis is done on one actual sample in each set of 20 samples. The MS/MSD will be fortified with the analyte of interest.

3.2 Recovery of the matrix spike are calculated using the following equation:

$$\text{Matrix spike percent recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spike sample results (µg/kg)
SR = Unfortified sample result (µg/kg)
SA = Spike added from spiking mix (µg/kg)

3.3 Calculate the relative percent difference (RPD) between the matrix spike and matrix spike duplicate using the following equation.

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

Where: D_1 = First sample % recovery
D_2 = Second sample % recovery (duplicate)

The RPD for each compound should not exceed 20%.

3.4 The matrix spike and matrix spike duplicate recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls inside the advisory limits of 70% to 130%.

4. Surrosate Spike Analysis

4.1 Prior to extraction, all samples, blanks, control and matrix spikes are fortified with the surrogate compound 3,4-dinitrotoluene to monitor the preparation, extraction and analysis efficiencies. The spiking level is 5,000 µg/kg.

4.2 Surrogate recoveries are calculated using the following equation:

$$\text{Surrogate spike percent recovery} = \frac{Qd}{Qa} \times 100$$

Where: Qd = Quantity determined by analysis (µg/kg)
Qa = Quantity added to sample (µg/kg)

4.3 The surrogate spike recovery must be evaluated by determining whether the concentration (measured as percent recovery) falls inside the advisory limits of 70% to 130%.

5. Nitroslycerin Identification

5.1 Before analyzing any samples, the analyst is required to determine the retention time for nitroglycerin.

5.2 Retention time windows for nitroglycerin will be established as ±0.14 from the continuing calibration.

5.3 UV Spectra is used for positive identification.

6. Initial Calibration

6.1 Standard solutions containing explosive analytes of interest are prepared at the five concentration levels described in the method. The five standard mixes are analyzed at the beginning of each run.

6.2 Tabulate the peak area response versus the concentration of the analyte in the standard. A response factor is calculated at each of the five levels as follows:

$$\text{Response factor (Rf)} = \frac{\text{Area Response}}{\text{Concentration of Analyte}}$$

6.3 Calculate the average response factor and percent relative standard deviation (%RSD) for each compound. The average response factor can be used for calculations if the response factor value is a constant over the working range of the detector [≤20 % RSD]. A minimum of five calibration standards will be used to determine the working range of the detector.

$$\%RSD = \frac{SD}{X} \times 100$$

Where SD =

$$STANDARD\ DEVIATION = \left| \sqrt{\frac{\sum_{i-1}^{n} (x_i - x)^2}{n-1}} \right|$$

7. Continuins Calibration

7.1 The average response factor must be verified after every ten sample injections and at the end of each run - by the measurement of the mid-level calibration standard. The response for the compound of interest must fall within ± 15% of the initial average response factor (% difference). If the parameter fails this test, a new initial calibration must be established. All samples run after a non-compliant continuing calibration standard must be reanalyzed with a new initial calibration.

CALCULATIONS:

1. Calculate the concentration of individual compounds in the sample using the following equation for external standards.

$$\text{Concentration } (\mu g/kg) = \frac{(A)*(V)*(D)*(Ca)}{(W)*(Rf)}$$

Where:

A = Area
Rf = Average response factor
V = Final extraction volume (L)
D = Analysis dilution factor of extract
Ca = Calcium chloride water dilution factor = 2
W = Weight of sample extracted (kg)

2. If the nitroglycerin area exceeds.the calibration range defined by the associated initial calibration, the respective samples must be diluted accordingly.

Method Detection Limits

Compound	MDL[a] (µg/kg)	EQL[1] (µg/kg)
Nitroglycerin	78	250

[a] Method Detection Limit determination, USEPA 40CFR Pt. 136, App. B, 1988.

[1] Estimated Quantitation Limit

En Chem, Inc.

Quality Assurance Document

SET No: 6

Standard Operating Procedure
Analytical Method

TITLE: Determination of Nitroaromatics and Nitramines in Soil/Sediment Samples by High Performance Liquid Chromatography (HPLC) -

DEPARTMENT: Semivolatile Organics

APPLICATION: This method covers the detenination of the following explosives in soils and sediments using High-Performance Liquid Chromatography (HPLC). The following compounds are analyzed under this method:

Compound	Abbreviation
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX
1,3,5-Trinitrobenzene	1,3,5-TNB
1,3-Dinitrobenzene	1,3-DNB
Methyl-2,4,6-trinitrophenylnitramine	Tetryl
Nitrobenzene	NB
2,4,6-Trinitrotoluene	2,4,6-TNT
4-Amino-2,6-dinitrotoluene	4-Amino-2,6-DNT
2-Amino-4,6-dinitrotoluene	2-Amino-4,6-DNT
2,4-Dinitrotoluene	2,4-DNT
2,6-Dinitrotoluene	2,6-DNT
2-Nitrotoluene	2-NT
3-Nitrotoluene	3-NT
4-Nitrotoluene	4-NT

PROCEDURE SUMMARY:

Homogenized portions of soil/sediment samples are extracted with acetonitrile in a chilled ultrasonic bath. The resulting extract is then taken through a 0.2 µm PTFE syringe filter ("acrodisc"). The explosives are uniquely separated by High-Performance Liquid Chromatography (HPLC) and the compounds of interest are identified and quantified by ultraviolet (UV) detection.

SENSITIVITY, PRECISION, AND ACCURACY:

The method detection limits (Attachment 1) represent the target detection limits that can be 'achieved in soil/sediment matrices using this method.
The precision, accuracy, and control limits for explosives in soil/sediment matrices are presented in Attachment 2.

REFERENCE:

U.S. Environmental Protection Agency (EPA). "Test Methods for Evaluating Solid Waste". SW-846. Method 8330. Revision 0 (September 1994).

U.S. Army Corps., Shell for Analytical Chemistry, 11/98.

REVIEWED BY: Daniel M. Rude 6-29-2000
Daniel M. Rude — Date
Semivolatile Group Leader

Gregory J. Graf 6-29-00
Gregory J. Graf — Date
Laboratory Operations

APPROVED BY: Glen A. Coder 6-29-2000
Glen A. Coder — Date
Laboratory Manager

SAFETY PRECAUTIONS:

The toxicity and carcinogenicity of the chemicals used in this method have not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

Standard precautionary measures used for handling other organic compounds should be sufficient for the safe handling of the analytes targeted by this method. Extra caution should be taken when handling the analytical neat material for the explosive itself and (in rare cases) where soil or waste samples are highly contaminated with the explosives.

Soil samples as high as 2% 2,4,6-TNT have been safely ground. Samples containing higher concentrations should not be ground in the mortar and pestle. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very fine ground grayish-white material.

Observe all standard laboratory safety procedures as outlined in the En Chem, Inc. Laboratory Safety Manual.

INTERFERENCES:

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample-processing hardware that may lead to discrete artifacts and/or elevated baselines in liquid chromatography. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending on the nature and diversity of the matrix involved. The cleanup procedure provided in this method may be used to minimize or overcome such interferences.

SAMPLE HANDLING AND STORAGE

Store samples and extracts at 4°C in the dark in Teflon-sealed containers.
Samples must be extracted within 14 days of sampling. Following extraction, the sample extracts must be analyzed within 40 days of extraction.

QUALITY ASSURANCE:

The minimum quality assurance/quality control operations necessary to satisfy the analytical requirements associated with the determination of the explosive compounds listed in this method are as follows:

1. Method Blank Analysis

1.1 A laboratory method blank is a sample of reagent sand that is carried through the entire analytical sequence (extraction, dilution, and analysis).

1.2 A method blank is analyzed with every 20 samples processed or whenever samples are extracted, whichever is most frequent. It is the analyst's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, and glassware are minimized. An acceptable laboratory method blank should contain less than the reporting limit of any single explosive target compound.

1.3 If a laboratory method blank exceeds the above criteria, the analyst must consider the analytical system to be out of control. The source of the contamination must be investigated, and appropriate corrective action must be taken, namely re-extraction of all associated samples with the contaminated method blank.

2. Control Spike Analysis

2.1 The control spike is a reagent sand sample which is fortified with the matrix spike solution. The control spike may be from the same source as the blank or from an independent source. All compounds of interest will be spiked.

2.2 A laboratory control spike is prepared and analyzed with every 20 samples processed or whenever samples are extracted, whichever is most frequent.

2.3 Recoveries of the laboratory control spike are calculated using the equation in Step 3.2.

2.4 The control spike recovery limits will be determined from the control spike recoveries. Interim control limits are included in Attachment 2.

NOTE: These limits are for advisory purposes only. These limits should not be used to determine if a sample should be re-extracted and reanalyzed.

2.5 To allow for sopradic failure, the following guidelines will be applied. One compound may be outside of control limits if there are 7-l 5 target analytes; 2 will be allowed if there are greater than 15 analytes. The recovery, however, must be greater than one half of the lower control limit for that analyte.

3. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

3.1 In order to evaluate the matrix effect of a sample on the analytical method and to provide both precision and accuracy information, an MS/MSD analysis is done on one

actual sample in each set of 20 samples. The MS/MSD will be fortified with all analytes of interest.

3.2 Individual component recoveries of the matrix spike are calculated using the following equation:

$$\text{Matrix spike percent recovery} = \frac{SSR - SR}{SA} \times 100$$

Where: SSR = Spike sample results (µg/kg)
SR = Unfortified sample result (µg/kg)
SA = Spike added from spiking mix (µg/kg)

3.3 Calculate the relative percent difference (RPD) between the matrix spike and matrix spike duplicate using the following equation.

$$RPD = \frac{C_1 - C_2 \times 100}{(C_1 + C_2)/2}$$

Where: C_1 = First sample concentration
C_2 = Second sample concentration (duplicate)

The RPD for each compound should not exceed 50%; 60% for Tetryl.

3.4 The matrix spike recovery limits will be determined from in-house data once 20-30 data-points have been collected. Interim control limits are included in Attachment 2.

NOTE: The limits in Attachment 2 are for advisory purposes only. These limits shoufd not be used to determine if a sample should be re-extracted and reanalyzed.

3.5 To allow for sopradic failure, the following guidelines will be applied. One compound may be outside of control limits if there are 7-15 target analytes; 2 will be allowed if there are greater than 15 analytes. The recovery, however, must be greater than one half of the lower control limit for that analyte.

4. Surrosate Spike Analysis

4.1 Prior to extraction, all samples, blanks, control and matrix spikes are fortified with the surrogate compound 3,4-dinitrotoluene to monitor the preparation, extraction and analysis efficiencies. The spiking level is 2,000 µg/kg.

4.2 Surrogate recoveries are calculated using the following equation:

$$\text{Surrogate spike percent recovery} = \frac{Qd}{Qa} \times 100$$

Where: Qd = Quantity determined by analysis (µg/kg)
Qa = Quantity added to sample (µg/kg)

4.3 The surrogate spike recovery must be evaluated by determining whether the concentration (measured as percent recovery) fails inside the advisory limits listed in Attachment 2.

NOTE: These limits were developed from a limited number of data points and are for advisory purposes only. These limits should not be used to determine if a sample should be reanalyzed.

5. Explosive Retention Time Windows

5.1 Before quantitating any samples, the analyst is required to determine the retention time windows for each explosive target compound. The retention time windows are used to identify explosive compounds during sample analysis.

5.2 Make at least three injections over a 72-hour period.

5.3 The width of ?he retention time window is defined as ± 3 standard deviations of the established mean. The minimum RT window will be ± 0.10 minute.

5.4 The center of the retention time window for each explosive compound is based on the retention time of the mid-point calibration standard from the corresponding ICAL.

5.5 All ongoing and ending standards will be used to monitor any retention time shifts of the compounds of interest. The retention time windows may be updated following successful calibration verification for the next group of samples analyzed. Each quantitation run must end with a mid-level calibration standard.

6. Initial Calibration

6.1 Standard solutions containing explosive analytes of interest are prepared at the six concentration levels describedin the method. The six standard mixes are analyzed at the beginning of each run.

6.2 Tabulate the peak area response versus the concentration of the analyte in the standard. A response factor for each analyte is calculated at each of the six levels as follows:

$$\text{Response factor (Rf)} = \frac{\text{Concentration of Analyte}}{\text{Peak Area Response}}$$

6.3 Calculate the average response factor and percent relative standard deviation (% RSD) for each compound. The average response factor can be used for calculations if the response factor value is a constant over the working range of the detector [≤20% RSD]. A minimum of five calibration standards will be used to determine the working range of the detector.

$$\%RSD = \frac{SD}{X} x100$$

Where SD = n-l Standard Deviation:

$$\sqrt{\frac{\sum_{i=1}^{n}(x_i - x)2}{n-1}}$$

6.4 Analysis of Initial Calibration Verification Standard: In order to consider the initial calibration acceptable, an Initial Calibration Verification Standard (ICV) must be analyzed within the same time clock as the calibration curve. The ICV standard must be from a second source stock and meet the same criteria as the Continuing Calibration Verification standard before the initial calibration may be considered valid.

7. Continuing Calibration

7.1 The average response factor must be verified after every twelve hours, however it is recommended that the verification be performed every ten sample injections and at the end of each run by the measurement of the mid-level calibration standard. The response for each compound of interest must fall within ± 15%D of the initial average response factor (30% D for Tetryl). If any parameter fails this test, a new initial calibration must be established. All samples run after a non-compliant continuing calibration standard must be reanalyzed with a new initial calibration.

7.2 Each target compound must be within the established RT window.

APPARATUS:

Volumetric pipette, 10 mL
Volumetric flasks, 10 mL, 25 mL
Volumetric syringes, 10 µL, 25 µL, 50 µL, 100 µL, 500 µL, and 1000 µL
Disposable glass pipettes
Beakers, assorted sizes, Griffin
20-mL amber glass vials, screw top sterile VOA vials with Teflon® -lined screw caps
12-mL amber vial test tube with Teflon@ -lined screw caps
Teflon@ or stainless steel spatulas
2-mL Luer Lock syringes
Vials, glass, 2-mL capacity with crimp top for LC autosampler

Vials, glass, 2-mL capacity with Teflon@ -lined screw cap for storage of stock standard, surrogate, and stock matrix spike solutions
Balance, Sartorius model L310
Ultrasonic Bath with chilling apparatus, Fisher Scientific model FS28 -
PTFE syringe filters, 0.2 pm, 25 mm diameter, Gelman Sciences, Part No. 4521
Analytical system complete with High-Performance Liquid Chromatograph and all required accessories including analytical column, gases, UV detector, recording integrator (a data system is required for measuring peak areas or peak heights and recording retention times).
HPLC System Consisting of:
-Gradient pumping system, Perkin-Elmer Corporation, Series 200 Pump
-5-u particle size diameter, 250 mm x 4.6 inner diameter, stainless steel column, Alltech BDS Cl 8 column, Alltech No. 1 1145
-5-u particle size diameter, 250 mm x 4.6 inner diameter, stainless steel column, Phenomenex Luna CN, catalog no. OOG-4255-EO
-Autoinjector, ISS-200, Perkin-Elmer
-Column heater, Perkin-Elmer LC oven Series 200
-Perkin-Elmer LC 235 Diode Array Detector or Perkin Elmer 785A UV-VIS
Aluminum weighing pan
Mortar and Pestle

NOTE: Equivalent equipment may be substituted.

REAGENTS:

Milli-Q® water brought to pH of 2-3 with Acetic Acid
Acetonitrile, HPLC grade
Methanol, HPLC grade
Sodium sulfate, granular, anhydrous, ACS, purified by heating at 400°C for 4 hours in a shallow tray
Calcium Chloride, reagent grade, J.T. Baker, Catalog No. 1311-01
Milli-Q® Water (reagent water)
Calcium Chloride/Water Solution - dilute 5 g of calcium chloride diluted to 1-L volume with Milli-Q® water
3,4-Dinitrotoluene, 1 .0 mg/mL in MeOH, AccuStandard or equivalent
14 component explosive mix, 1 .0 mg/mL in MeOH:ACN (1 :1), AccuStandard or equivalent
Ottawa sand, Fisher, purified by heating at 400°C for 4 hours in a shallow tray
Acetic Acid, Baxter

NOTE: Equivalent reagents may be substituted.

STANDARD & SPIKING SOLUTION PREPARATION:

Preoaration of Explosive Workino Calibration Stock Solution

Prepare a IO µg/mL explosive working stock solution by diluting 100 µL of a 1 .O mg/mL commercially available 14-component explosive mix stock ampule and 100 µL of 1 .O mg/mL commercially available 3,4-DNT (surrogate) stock ampule. Bring to 10 mL final volume in a 10mL volumetric flask with HPLC grade acetonitrile.

Preparation of Explosive Analyte Calibration Standards

1. Calibration standards are prepared at six concentration levels from the explosive working calibration stock solution. The concentrations listed below reflect the effective concentration **prior** to diluting each standard with pH adjusted Milli-Q® water or $CaCl_2$ solution.

1.1 To prepare Calibration Standard 40 µg/L, dilute 40 µL of the IO µg/mL explosive working calibration stock solution volumetrically to mark in 10 mL of acetonitrile.

1.2 To prepare Calibration Standard 100 µg/L, dilute 100 µL of the 10 µg/mL explosive working calibration stock solution volumetrically to mark in 10 mL of acetonitrile.

1.3 To prepare Calibration Standard 200 µg/L, dilute 200 µL of the 10 µg/mL explosive working calibration stock solution volumetrically to mark in 10 mL of acetonitrile.

1.4 To prepare Calibration Standard 300 µg/L, dilute 300 µL of the 10 µg/mL explosive working calibration stock solution volumetrically to mark in 10 mL of acetonitrile.

1.5 To prepare Calibration Standard 400 µg/L, dilute 400 µL of the 10 µg/mL explosive working calibration stock solution volumetrically to mark in 10 mL of acetonitrile.

1.6 To prepare Calibration Standard 500 µg/L, dilute 500 µL of the 10 µg/mL explosive working calibration stock standard volumetrically to mark in 10 mL of acetonitrile.

2. Just prior to analysis, 400-µL of each of the six working calibration standards are mixed with 1000-µL of the calcium chloride/water solution OR Milli-Q® water adjusted to pH 2-3 with acetic acid in a 2-mL GC crimp top vial. When the solutions are mixed thoroughly they are ready for injection.

NOTE: All calibration standards (prepared by diluting with calcium chloride/water solution OR Milli-Q® water adjusted to pH 2-3 with acetic acid) must be prepared fresh daily.

Preparation of 14-component Explosive Spiking Solution @ 100 µg/mL :

Dilute 1 .O mL of 1.0 mg/mL of commercially prepared 14-component explosive mix volumetrically to mark in 10 mL of methanol.

NOTE: Prepare the Explosive Standards Stock Solution and the Explosive Spiking Solution from either different lots or vendors of stock 14-component explosive mix used for calibration standards.

Preparation of Surrogate Spiking Solution @ 100 µg/mL :

Dilute 1 .O mL of 1 .O mg/mL of commercially prepared 3,4-Dinitrotoluene volumetrically to mark in 10 mL of methanol.

PROCEDURE:

Sample Homoaenization

1. Dry soil samples in a hood, at room temperature or colder, to a constant weight. Do this by placing approximately 20 to 30 grams of a well mixed subsample of wet sample to an aluminum weighing pan. Place the weighing pans with the sample under the hood and allow to air dry for approximately 24 hours. Do not to expose the samples to direct sunlight.

2. Grind and homogenize the dried samptes thoroughly in an acetonitrile-rinsed mortar to pass a 30 mesh sieve.

Sample Extraction

1. Weigh 2.0 g of sand into a 20-mL VOA vial; this will represent the method blank.

2. Weigh 2.0 g of the sand into a second 20-mL VOA vial; this will be used for the control spike.

3. Weigh 2.0 g of each sample and MSNSD into a 20-mL VOA vial.

4. Spike 40 µL of the 100 µg/mL 3,4-dinitrotoluene (surrogate) solution into each 20-mL vial.

5. Spike 40 µL of the 100 µg/mL explosive spiking solution into the control spike, matrix spike and matrix spike duplicate.

6. Add 10 mL of HPLC grade acetonitrile via volumetric pipette to each 20-mL vial.

7. Cap each of the 20-mL amber VOA vials and manually shake each vial for 2 minutes.

8. Place vials in a chilled ultrasonic bath and sonicate for 18 hours without exception.

9. Remove vials from ultrasonic bath and allow sample particulate to settle and equilibrate to room temperature for at least 20 minutes.

10. Filter 2 mL of the supernatant extract through a 0.2 pm PTFE syringe filter (acrodisc) using a 2 mL Luer Lock syringe into an 5-mL culture tube.

11. Combine 400 µL of the filtered extract with 1000 µL of 5 g/L calcium chloride water or Milli-Q® water adjusted to pH 2-3 with acetic acid directly in an injection vial. Store the unused portion of the filtered extract in the refrigerator.

12. The sample extract is ready for HPLC analysis.

Recommended HPLC Materials and Operating Conditions

ANALYTICAL COLUMN: Alltech BDS Cl8 column, 5-µ particle size diameter, 250 mm x 4.6 mm inner diameter, stainless steel column, Alltech No. 11145

CONFIRMATION COLUMN: Phenomenex Luna CN, 5-u particle size diameter, 250 mm x 4.6 mm inner diameter, Phenomenex No. OOG-4255-EO

GUARD COLUMN: 4 mm C-l 8 packing Phenomenex No. AJO-4287

CONFIRMATION GUARD COLUMN: 4 mm CN packing Phenomenex No. AJO-4305

ANALYTICAL COLUMN PUMP PARAMETERS: Flow rate: 1 .O mL/minute gradient, with Methanol and Milli-Q® water:

MeOH:Water

Step 0. 30:70 for 5 minute equilibration
Step 1. 45:55 in 15 minutes
Step 2. 50:50 in 15 minutes
Step 3. 60:40 in 5 minutes

Total run time, less equilibration time: 35 minutes.

AUTO SAMPLER:

Sample loop: 200 µL
Volume injected: 200 µL

COLUMN TEMPERATURE:
35 ° c

DETECTOR SETTINGS:

UV wavelength: 250nm
Sensitivity: 0.0005

GAS REQUIRED:

Helium 99.99+ percent purity for degassing mobile phase.

CALCULATIONS:

1. Calculate the concentration of individual compounds in the sample using the following equation for external standards:

$$\text{Concentration } (\mu g/Kg) = \frac{(A)*(Rf)*(V)*(D)*}{(W)}$$

Where:

A = Area
Rf= Average response factor (µg/L)
v = Final extraction volume (L)
D = Analysis dilution factor of extract
w = Weight of sample extracted (Kg)

NOTE: Dilution prior to analyzing with Milli-Q® water adjusted to pH 2-3 with acetic acid or $CaCl_2$ water is canceled bycalibration standards being diluted in the same fashion prior to analysis.

2. If an explosive target compound area exceeds the calibration range defined by the associated initial calibration, the respective samples must be diluted accordingly.

CONFIRMATION:

All compounds found above their respective quantitation limits will be confirmed by analysis on a cyano column using the same analysis procedures and instrument parameters, with the exception of a cyano column and the following pump parameters:

Flow rate: 2.0 mL/minute, Gradient with acetonitrile and Milli-Q® water:

ACN:Water

Step 0. 10:90 for 5 minute equilibration
Step 1. 20:80 in 5 minutes
Step 2. 80:20 in IO minutes

Total run time, less equilibration time: 15 minutes

The confirmation analysis is for qualitative use only. The analyte is considered confirmed if it falls within its respective RT window on the cyano column.

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

Attachment 1

Method Detection Limits
Explosive Residues in Soil Matrix

Compound	EQL (µg/Kg)
HMX	200
RDX	200
1,3,5-Trinitrobenzene	200
1,3-Dinitrobenzene	200
Tetryi	200
Nitrobenzene	200
2,4,6-Trinitrotoluene	200
4-Amino-2,6-dinitrotoluene	200
2-Amino-4,6-dinitrotoluene	200
2,4-Dinitrotoluene	200
2,6-Dinitrotoluene	200
2-Nitrotoluene	200
3-Nitrotoluene	200
4-Nitrotoluene	200

NOTE: 200 µg/Kg has been determined to be the lowest reporting limit achievable keeping an appropriate signal to noise ratio on the cyano column comfirmation analysis; although all calculated MDLs, with the exception of Tetryl, are considerably lower.

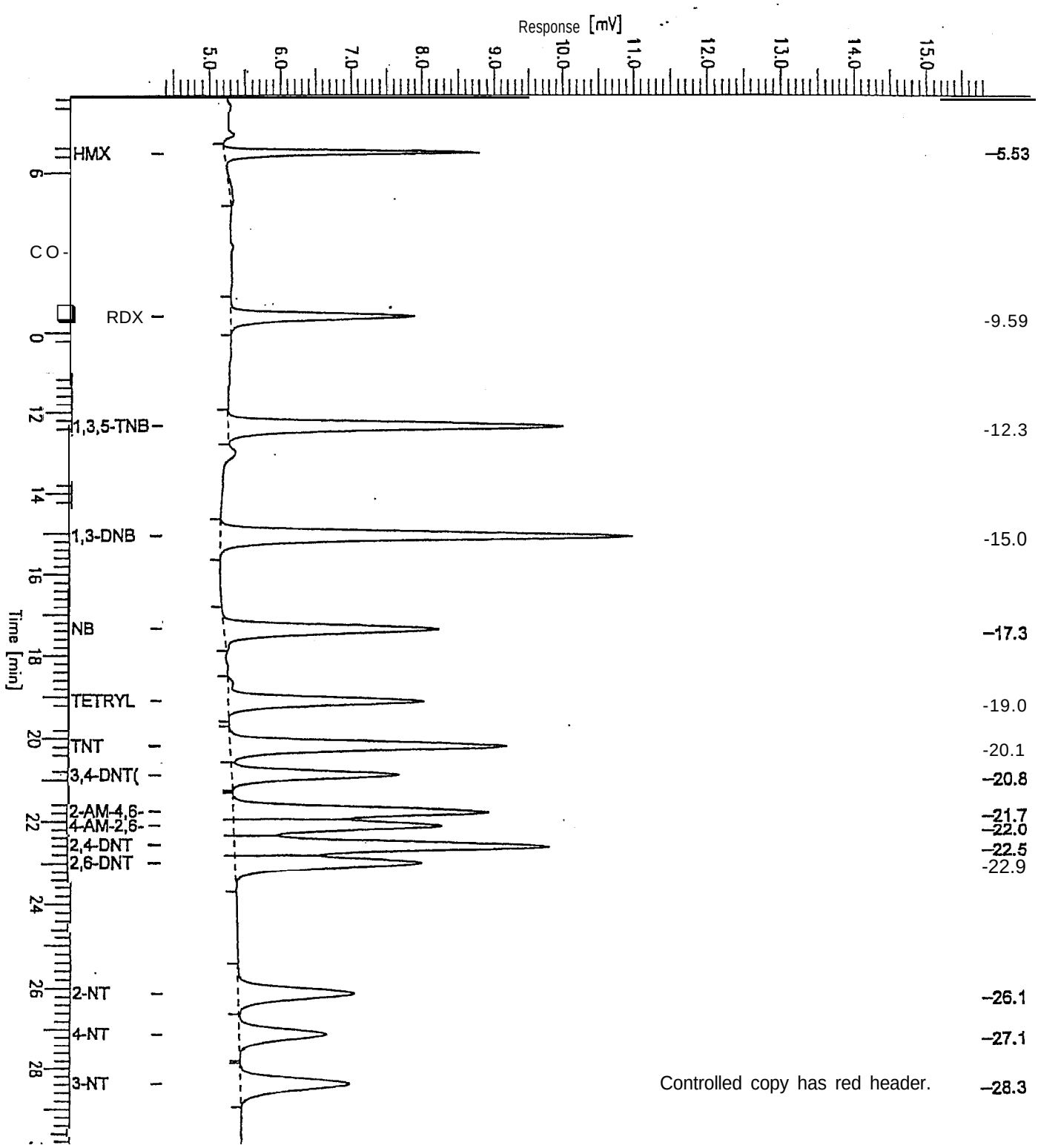
Attachment 2

Precision and Accuracy
Explosive Residues
Soil Matrix

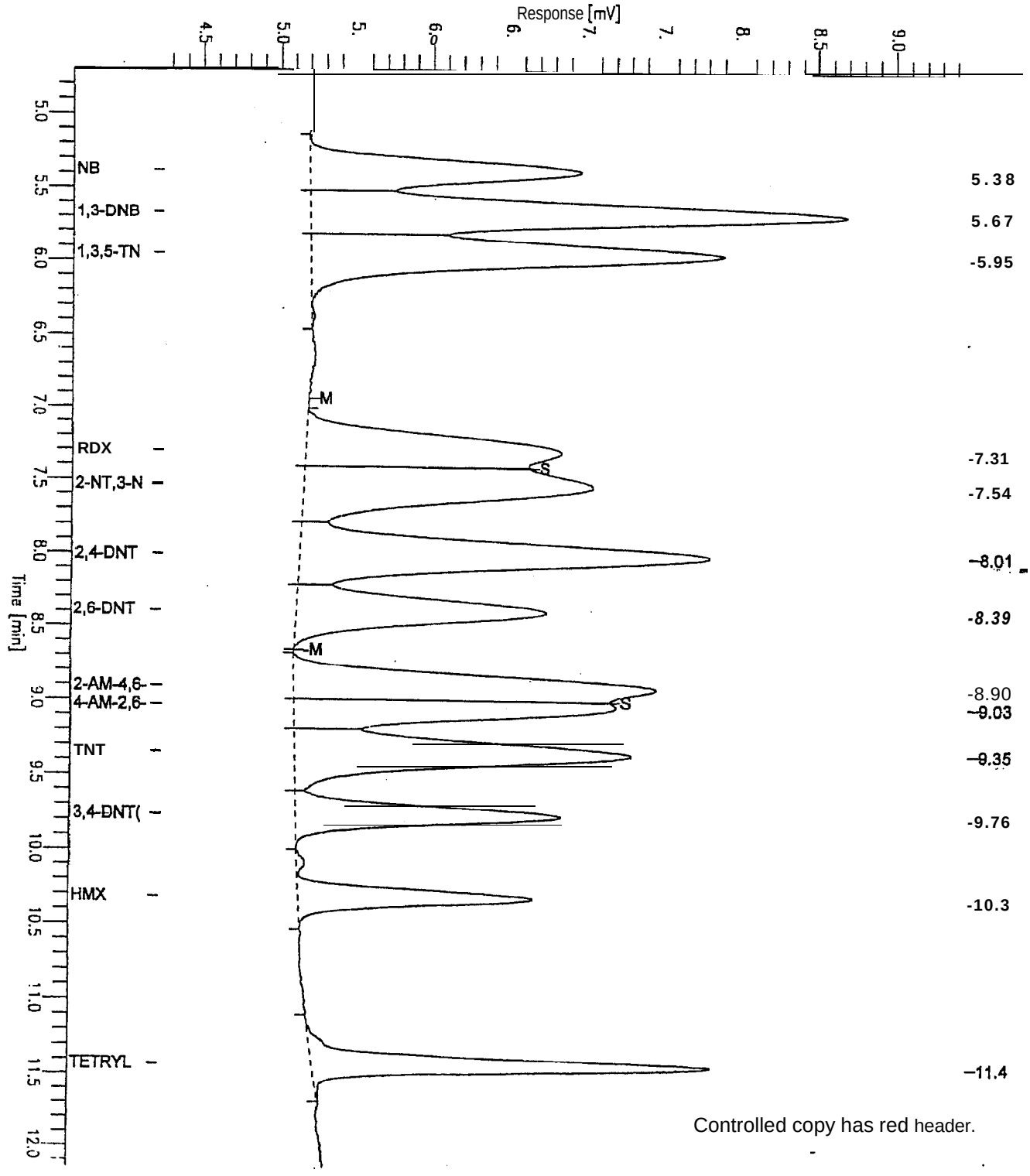
Compound	Matrix Spike Limits (%)	Control Spike Limits (%)
HMX	50 - 140	60 - 120
RDX	50 - 140	60-120
1,3,5-Trinitrobenzene	50 - 140	60 - 120
1,3-Dinitrobenzene	50 - 140	60 - 120
Tetryl	45 - 145	45 - 145
Nitrobenzene	50 - 140	60 - 120
2,4,6-Trinitrotoluene	50 - 140	60-120
4-Amino-2,6-DNT	50 - 140	60-120
2-Amino-4,6-DNT	50 - 140	60 - 120
2,4-Dinitrotoluene	50 - 140	60 - 120
2,6-Dinitrotoluene	50 - 140	60 - 120
2-Nitrotoluene	50 - 140	60 - 120
3-Nitrotoluene	50 - 140	60 - 120
4-Nitrotoluene	50 - 140	60-120
3,4-DNT (surrogate)	50 - 150	50 - 150

Advisory control limits are to be used until a sufficient number of points are generated to calculate in-house limits. The limits in use may differ from those listed.

Attachment 3
Example Chromatogram
Alltech BDS Cl8 Column



Attachment 4
Example Chromatogram
Phenomenex Luna CN



EN CHEM MADISON LABORATORY ANALYTICAL METHODS, QA OBJECTIVES FOR NON-AQUEOUS SAMPLES											
		Effective June 2001 to June 2002							5/4/01		
Parameter	Units	Method	Method	LCS	LCS	Accuracy	Precision	MDL	Reporting	W. Length	En Chem
		(prep)	(analytical)	(% Rec.)	(% RPD)	(% Rec.)	(% RPD)		Limit	Alt.	SOP #
										Isotope	
HPLC 8330											
1,3 - Dinitrobenzene	ug/kg	8330	8330	60-120	N/A	50-140	0-50	19.31	200	N/A	SVO-69
1,3,5 - Trinitrobenzene	ug/kg	8330	8330	60-120	N/A	50-140	0-50	25.28	200	N/A	SVO-69
2 - Amino - 4,6 - dinitrotoluene	ug/kg	8330	8330	60-120	N/A	50-140	0-50	24.41	200	N/A	SVO-69
2 - Nitrotoluene	ug/kg	8330	8330	60-120	N/A	50-140	0-50	36.28	200	N/A	SVO-69
2,4 - Dinitrotoluene	ug/kg	8330	8330	60-120	N/A	50-140	0-50	20.55	200	N/A	SVO-69
2,4,6 - Trinitrotoluene	ug/kg	8330	8330	60-120	N/A	50-140	0-50	38.40	200	N/A	SVO-69
2,6 - Dinitrotoluene	ug/kg	8330	8330	60-120	N/A	50-140	0-50	19.55	200	N/A	SVO-69
3 - Nitrotoluene	ug/kg	8330	8330	60-120	N/A	50-140	0-50	16.88	200	N/A	SVO-69
4 - Amino - 2,6 - dinitrotoluene	ug/kg	8330	8330	60-120	N/A	50-140	0-50	42.19	200	N/A	SVO-69
4 - Nitrotoluene	ug/kg	8330	8330	60-120	N/A	50-140	0-50	34.23	200	N/A	SVO-69
HMX	ug/kg	8330	8330	60-120	N/A	50-140	0-50	39.71	200	N/A	SVO-69
Nitrobenzene	ug/kg	8330	8330	60-120	N/A	50-140	0-50	19.68	200	N/A	SVO-69
RDX	ug/kg	8330	8330	60-120	N/A	50-140	0-50	21.67	200	N/A	SVO-69
Tetryl	ug/kg	8330	8330	45-145	N/A	45-145	0-60	112.72	200	N/A	SVO-69

Effective June 2001 to June 2002

Standard Ooeratina Procedure - Analytical Method

TITLE: Determination of Nitroaromatics and Nitramines in Aqueous Samples by High Performance Liquid Chromatography (HPLC) - .

DEPARTMENT: Semivolatile Organics

APPLICATION: This method covers the determination of the following explosive compounds in aqueous samples using High-Performance Liquid Chromatography (HPLC). The following compounds are analyzed under this method:

Comoound	Abbreviation
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX
1,3,5-Trinitrobenzene	1,3,5-TNB
1,3-Dinitrobenzene	1,3-DNB
Methyl-2,4,6-trinitrophenylnitramine	Tetryl
Nitrobenzene	NB
2,4,6-Trinitrotoluene	2,4,6-TNT
4-Amino-2,6-dinitrotoluene	4-Amino-2,6-DNT
2-Amino-4,6-dinitrotoluene	2-Amino-4,6-DNT
2,4-Dinitrotoluene	2,4-DNT
2,6-Dinitrotoluene	2,6-DNT
2-Nitrotoluene	2-NT
3-Nitrotoluene	3-NT
4-Nitrotoluene	4-NT

PROCEDURE SUMMARY:

Water samples are extracted via solid phase extraction columns. The resulting extract is then taken through a 0.2 µm PTFE syringe filter ("acrodisc"). The explosives are uniquely separated by High-Performance Liquid Chromatography (HPLC) and the compounds of interest are identified and quantified by ultraviolet detection.

SENSITIVITY AND QC LIMITS:

The method detection limits (Attachment 1) represent the target detection limits that can be achieved in aqueous matrices using this method.
The control limits for explosives in aqueous matrices are presented in Attachment 2.

REFERENCES:

U.S. Environmental Protection Agency (EPA). "Test Methods for Evaluating Solid Waste". SW-846. Method 8330. Revision 0 (September 1994).
U.S. Army Corps., Shell for Analytical Chemistry, 11/98.

REVIEWED BY: _____ 11/9/2000
Daniel M. Rude Date
Semivolatiles Group Leader

_____ 11/9/00
Gregory J. Graf Date
Quality Assurance Officer

APPROVED BY: Gregory J. Graf, for 11/9/00
Glen A. Coder Date
Laboratory Manager

SAFETY PRECAUTIONS:

The toxicity and carcinogenicity of the chemicals used in this method have not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Laboratory staff should observe‘all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

Standard precautionary measures used for handling other organic compounds should be sufficient for the safe handling of the analytes targeted by this method. Extra caution should be taken when handling the analytical neat material for the explosive itself and (in rare cases) where soil or waste samples are highly contaminated with the explosives.

Observe all standard laboratory safety procedures as outlined in the En Chem, Inc. Laboratory Safety Manual.

INTERFERENCES:

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample-processing hardware that may lead to discrete artifacts and/or elevated baselines in liquid chromatography. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending on the nature and diversity of the matrix involved. The cleanup procedure provided in this method may be used to minimize or overcome such interferences.

SAMPLE HANDLING AND STORAGE

Store samples and extracts at 4°C in the dark in Teflon-sealed containers.
Samples must be extracted within 7 days of sampling. Following extraction, the sample extracts must be analyzed within 40 days of extraction.

QUALITY ASSURANCE:

The minimum quality assurance/quality control operations necessary to satisfy the analytical requirements associated with the determination of the explosive compounds listed in this method are as follows:

1. Method Blank Analysis

1.1 A laboratory method blank is a sample of Milli-Q® water that is carried through the entire analytical sequence (extraction, dilution, and analysis).

1.2 A method blank is analyzed with every 20 samples processed or whenever samples are extracted, whichever is most frequent. It is the analyst’s responsibility to

ensure that method interferences caused by contaminants in solvents, reagents, and glassware are minimized. An acceptable laboratory method blank should contain less than the reporting limit of any single explosive target compound.

1.3 If a laboratory method blank exceeds the above criteria, the analyst must consider the analytical system to be out of control. The source of the contamination must be investigated, and appropriate corrective action must be taken, namely re-extraction of all associated samples with the contaminated method blank.

2. Control Spike 'Analysis

2.1 The control spike is a Milli-Q® water sample which is fortified with the matrix spike solution. The control spike may be from the same source as the blank or from an independent source. All compounds of interest will be spiked.

2.2 A laboratory control spike is prepared and analyzed with every 20 samples processed or whenever samples are extracted, whichever is most frequent.

2.3 Recoveries of the laboratory control spike are calculated using the equation in Step 3.2.

2.4 The control spike recovery limits will be determined from the control spike recoveries. Interim control limits are included in Attachment 2.

NOTE: These limits are for advisory purposes only. These limits should not be used to determine if a sample should be re-extracted and reanalyzed.

2.5 To allow for sopradic failure, the following guidelines will be applied. One compound may be outside of control limits if there are 7-l 5 target analytes; 2 will be allowed if there are greater than 15 analytes. The recovery, however, must be greater than one half of the lower control limit for that analyte.

3. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

3.1 In order to evaluate the matrix effect of a sample on the analytical method and to provide both precision and accuracy information, an MS/MSD analysis Is done on one actual sample in each set of 20 samples. The MS/MSD will be fortified with all analytes of interest.

3.2 Individual component recoveries of the matrix spike are calculated using, the following equation:

$$\text{Matrii spike percent recovery} = \frac{SSR - SR}{SA} \times 100$$

Where: SSR = Spike sample results (µg/L)
SR = Unfortified sample result (µg/L)
SA = Spike added from spiking mix (µg/L)

3.3 Calculate the relative percent difference (RPD) between the matrix spike and matrix spike duplicate using the following equation.

$$RPD = \frac{C_1 - C_2 \times 100}{(C1 + C_2)/2}$$

Where: C_1 = First sample concentration
C_2 = Second sample concentration (duplicate)

The RPD for each compound should not exceed 50%; 60% far Tetryl.

3.4 The matrix spike recovery limits will be determined from in-house data once 20-30 data-points have been collected. interim control limits are included in Attachment 2.

NOTE: The limits in Attachment 2 are for advisory purposes only. These limits should not be used to determine if a sample should be re-extracted and reanalyzed.

3.5 To allow for sopradic failure, the following guidelines will be applied. One compound may be outside of control limits if there are 7-15 target analytes; 2 will be allowed if there are greater than 15 analytes. The recovery, however, must be greater than one half of the lower control limit for that analyte.

4. Surroaate Spike Analvsis

4.1 Prior to extraction, all samples, blanks, control and matrix spikes are fortified with the surrogate compound 3,4-dinitrotoluene to monitor the preparation, extraction and analysis efficiencies. The spiking level is 4.0 µg/L.

4.2 Surrogate recoveries are calculated using the following equation:

$$\text{Surrogate spike percent recovery} = \frac{Qd}{Qa} \times 100$$

Where: Qd = Quantity determined by analysis (µg/L)
Qa = Quantity added to sample (µg/L)

4.3 The surrogate spike recovery must be evaluated by determining whether the concentration (measured as percent recovery) falls inside the advisory limits listed in Attachment 2.

NOTE: These limits were developed from a limited number of data points and are for advisory purposes only. These limits should not be used to determine if a sample should be reanalyzed.

5. Explosive Retention Time Windows

5.1 Before quantitating any samples, the analyst is required to determine the retention time windows for each explosive target compound. The retention time windows are used to identify explosive compounds during sample analysis.

5.2 Make at least three injections over a 72-hour period.

5.3 The width of the retention time window is defined as ± 3 standard deviations of the established mean. The minimum RT window will be ± 0.10 minute.

5.4 The center of the retention time window for each explosive compound is based on the retention time of the mid-point calibration standard from the corresponding ICAL.

5.5 All ongoing and ending standards will be used to monitor any retention time shifts of the compounds of interest. The retention time windows may be updated following successful calibration verification for the next group of samples analyzed. Each quantitation run must end with a mid-level calibration standard.

6. Initial Calibration

6.1 Standard solutions containing explosive analytes of interest are prepared at the six concentration levels described in the method. The six standard mixes are analyzed at the beginning of each run.

6.2 Tabulate the peak area response versus the concentration of the analyte in the standard. A response factor for each analyte is calculated at each of the six levels as follows:

$$\text{Response factor (Rf)} = \frac{\text{Concentration of Analyte}}{\text{Peak Area Response}}$$

6.3 Calculate the average response factor and percent relative standard deviation (% RSD) for each compound. The average response factor can be used for calculations if the response factor value is a constant over the working range of the detector [≤20% RSD]. A minimum of five calibration standards will be used to determine the working range of the detector.

$$\%RSD = \frac{SD}{X} x100$$

Where SD = n-l Standard Deviation:

$$\left|\sqrt{\frac{\sum_{i=1}^{n}(x_i - x)2}{n-1}}\right|$$

6.4 Analysis of Initial Calibration Verification Standard: In order to consider the initial calibration acceptable, an Initial Calibration Verification Standard (ICV) must be analyzed within the same time clock as the calibration curve. The ICV standard must be from a second source stock and meet the same criteria as the Continuing Calibration Verification standard before the initial calibration may be considered valid.

7. Continuinn Calibration

7.1 The average response factor must be verified after every twelve hours, however it is recommended that the verification be performed every ten sample injections and at the end of each run by the measurement of the mid-level calibration standard. The response for each compound of interest must fall within ± 15%D of the initial average response factor (30% D for Tetryl). If any parameter fails this test, a new initial calibration must be established. All samples run after a non-compliant continuing calibration standard must be reanalyzed with a new initial calibration.

7.2 Each target compound must be within the established RT window.

APPARATUS:

Volumetric flasks, 5.0 mL,10 mL, 25 mL
Volumetric syringes, 10 µL, 25 µL, 50 µL, 100 µL, 500 µL, and 1000 µL
Disposable glass pipettes
1000 mL graduated cylinder
1000 mL or 500 mL amber glass sample jar

10 mL amber vial with Teflon@ -lined screw caps
2-mL Luer Lock syringes
Vials, glass, 2-mL capacity with crimp top for LC autosampler
Vials, glass, 10-mL capacity with Teflon@ -lined screw cap for storage of stock standard, surrogate, and stock matrix spike solutions
J.T. Baker SPE-12G, vacuum manifoid
Varian, Bond Elut solid phase extraction columns, 6-mL, 500mg of ENV bonded phase, 80-100 mesh
Vacuum pump
Aspirator
Visiprep large volume sampler tubing
Vacuum manifold trap kit
PTFE syringe filters, 0.2 pm, 25 mm diameter, Gelman Sciences, Part No. 4521

Analytical system complete with High-Performance Liquid Chromatograph and all required accessories including analytical column, gases, UV detector, recording integrator (a data system is required for measuring peak areas or peak heights and recording retention times).
HPLC System Consisting of:
-Gradient pumping system, Perkin-Elmer Corporation, Series 200 Pump
-5-u particle size diameter, 250 mm x 4.6 inner diameter, stainless steel column,' Alltech BDS Cl 8 column, Alltech No. 11145
-5-u particle size diameter, 250 mm x 4.6 inner diameter, stainless steel column, Phenomenex Luna CN, catalog no. OOG-4255-EO
-Autoinjector, ISS-200, Perkin-Elmer
-Column heater, Perkin-Elmer LC oven Series 200
-Perkin-Elmer LC 235 Diode Array Detector or Perkin Elmer 785A UV-VIS

NOTE: Equivalent equipment may be substituted.

REAGENTS:

Milli-Q® water brought to pH of 2-3 with Acetic Acid
Acetonitrile, HPLC grade
Methanol, HPLC grade
Calcium Chloride, reagent grade, J.T. Baker, Catalog No. 131 I-01
Milli-Q® Water (reagent water)
Calcium Chloride/Water Solution - dilute 5 g of calcium chloride diluted to 1-L volume with Milli-Q® water
3,4-Dinitrotoluene, 1.O mg/mL in MeOH, AccuStandard or equivalent
14 component explosive mix, 1.0 mg/mL in MeOH:ACN (1:1), AccuStandard or equivalent
Acetic Acid, Baxter

NOTE: Equivalent reagents may be substituted.

STANDARD & SPIKING SOLUTION PREPARATION:

Preoaration of Explosive Workina Calibration Stock Solution

Prepare a 10 µg/mL explosive working stock solution by diluting 100 µL of a 1 .O mg/mL commercially available 14-component explosive mix stock ampule and 100 µL of 1 .O mg/mL commercially available 3,4-DNT (surrogate} stock ampule. Bring to 10 mL final volume in a 10 mL volumetric flask with HPLC grade acetonitrile.

Preparation of Explosive Analyte Calibration Standards

1. Calibration standards are prepared at six concentration levels from the explosive working calibration stock solution. The concentrations listed below reflect the effective concentration **prior** to diluting each standard with pH adjusted Milli-Q® water or $CaCl_2$ solution.

1.1 To prepare Calibration Standard 40 µg/L, dilute 40 µL of the 10 µg/mL explosive working calibration stock solution volumetrically to mark in 10 mL of acetonitrile.

1.2 To prepare Calibration Standard 100 µg/L, dilute 100 µL of the 10 µg/mL explosive working calibration stock solution volumetrically to mark in 10 mL of acetonitrile.

1.3 To prepare Calibration Standard 200 µg/L, dilute 200 µL of the 10 µg/mL explosive working calibration stock solution volumetrically to mark in 10 mL of acetonitrile.

1.4 To prepare Calibration Standard 300 µg/L, dilute 300 µL of the 10 µg/mL explosive working calibration stock solution volumetrically to mark in 10 mL of acetonitrile.

1.5 To prepare Calibration Standard 400 µg/L, dilute 400 µL of the 10 µg/mL explosive working calibration stock solution volumetrically to mark in 10 mL of acetonitrile.

1.6 To prepare Calibration Standard 500 µg/L, dilute 500 µL of the 10 µg/mL explosive working calibration stock standard volumetrically to mark in 10 mL of acetonitrile.

2. Just prior to analysis, 400-µL of each of the six working calibration standards are mixed with 1000-µL of the calcium chloride/water solution OR Milli-Q® water adjusted to pH 2-3 with acetic acid in a 2-mL GC crimp top vial. When the solutions are mixed thoroughly they are ready for injection.

NOTE: All calibration standards (prepared by diluting with calcium chloride/water solution OR Milli-Q® water adjusted to pH 2-3 with acetic acid) must be prepared fresh' daily.

Preparation of 14-component Explosive Spiking Solution @ 100 µg/mL :

Dilute 1 .O mL of 1 .O mg/mL of commercially prepared 14component explosive mix volumetrically to mark in 10 mL of methanol.

NOTE: Prepare the Explosive Standards Stock Solution and the Explosive Spiking Solution from either different lots or vendors of stock 14-component explosive mix used for calibration standards.

Preparation of Surroaate Soikina Solution @ 100 µg/mL :

Dilute 1 .O mL of 1 .O mg/mL of commercially prepared 3,4-Dinitrotoluene volumetrically to mark in 10 mL of methanol.

PROCEDURE:

SPE Tube Prep

1. Place the SPE columns into flow control valves atop the vacuum manifold.

2. Place appropriate end of the color-coded Visiprep tubing into the SPE columns and the other end into a beaker of acetonitrile.

3. Attach vacuum manifold to waste trap flask and attach flask to vacuum pump.

4. Open all flow control valves and turn on pump making sure the vacuum is controlled so the flow rate through the columns is less than 5 mLs a minute. At this time make sure all the columns have are flowing at approximately 5 mL/min. by adjusting the flow control values.

5. After 5 minutes turn the vacuum pump off and transfer the sample lines from the beaker of acetonitrile to a beaker of Milli-Q® water. Turn the pump back on making sure the flow rate for column is approximately 5 mLs/min. by adjusting the flow control valves under each column. After 10 minutes turn the pump off leaving the sample lines in the beaker of water.

Sample Extraction

1. Invert sample container three times and then transfer 500 mL to a prelabeled 1000 mL or 500 mL glass amber sample jar.

2. Spike 20 µL of the 100 µg/mL 3,4-dinitrotoluene (surrogate) solution into each 500-mL sample.

3. Spike 20 µL of the 100 µg/mL explosive spiking solution into the control spike, matrix spike and matrix spike duplicate.

4. Label each SPE column with a corresponding sample number and place the sampler line connected to it into the 500 mLs of sample. Repeat this for each sample.

5. Attach the vacuum manifold to the aspirator .

6. Turn on the water for the aspirator and adjust each SPE column to approximately 10 mL/min using the control values. The sample container should be tilted and the sample tubing should be placed at the lowest point to insure all 500 mL is put through the column.

7. After the full 500 mL of sample has gone through the column let the vacuum continue to insure as much of the water as possible is removed and column appears dry.(2-3 min.).

8. Remove the sampler tubing from the top of each column and the place a prelabeled 5 mL volumetric under it's matching SPE column in the manifold.

9. Put 5 mL of ACN in each SPE tube. Turn aspirator on and elute into 5 mL volumetric at a flow rate of approximately 3 mLs/min.

10 When all columns are done dripping remove them from the manifold and adjust them to the 5.0 mL line on the volumetric with ACN.

10. Filter all of the supernatant extract through a 0.2 pm PTFE syringe filter (acrodisc) using a 2 mL Luer Lock syringe into an prelabeled 5-mL amber screw top vial.

11. Combine 400 µL of the filtered extract with 1000 µL of 5 g/L calcium chloride water or Milli-Q® water adjusted to pH 2-3 with acetic acid directly in an injection vial. Store the unused portion of the filtered extract in the refrigerator.

12. The sample extract is ready for HPLC analysis.

Recommended HPLC Materials and Operating Conditions

ANALYTICAL COLUMN: Alltech BDS Cl 8 column, 5-u particle size diameter, 250 mm x 4.6 mm inner diameter, stainless steel column. Alltech No. 11145

CONFIRMATION COLUMN: Phenomenex Luna CN, 5-p particle size diameter, 250 mm x 4.6 mm inner diameter, Phenomenex No. OOG-4255-EO

GUARD COLUMN: 4 mm C-18 packing Phenomenex No. AJO-4287

CONFIRMATION GUARD COLUMN: 4 mm CN packing Phenomenex No. AJO-4305

ANALYTICAL COLUMN PUMP PARAMETERS: Flow rate: 1 .O mL/minute gradient, with Methanol and Milli-Q® water:

MeOH:Water

Step 0. 30:70 for 5 minute equilibration
Step 1. 45:55 in 15 minutes
Step 2. 50:50 in 15 minutes
Step 3. 60:40 in 5 minutes

Total run time, less equilibration time: 35 minutes.

AUTO SAMPLER:

Sample loop: 200 µL
Volume injected: 200 µL

COLUMN TEMPERATURE:
35 ° c

DETECTOR SETTINGS:

UV wavelength: 250nm
Sensitivity: 0.0005

GAS REQUIRED:

Helium 99.99+ percent purity for degassing mobile phase.

CALCULATIONS:

1. Calculate the concentration of individual compounds in the sample using the following equation for external standards:

$$\text{Concentration } (\mu g/Kg) = \frac{(A)*(Rf)*(V)*(D)*}{(W)}$$

Where:

A = Area
Rf= Average response factor (µg/L)
v = Final extraction volume (L)
D = Analysis dilution factor of extract
w = Weight of sample extracted (Kg)

NOTE: Dilution prior to analyzing with Milli-Q® water adjusted to pH 2-3 with acetic acid or $CaCl_2$ water is canceled by calibration standards being diluted in the same fashion prior to analysis.

2. If anexplosive target compound area exceeds the calibration range defined by the associated initial calibration, the respective samples must be diluted accordingly.

CONFIRMATION:

All compounds found above their respective quantitation limits will be confirmed by analysis on a cyano column using the same analysis procedures and instrument parameters, with the exception of a cyano column and the following pump parameters:

Flow rate: 2.0 ml/minute, Gradient with acetonitrile and Milli-Q® water:

ACN:Water

Step 0. 10:90 for 5 minute equilibration
Step 1. 20:80 in 5 minutes
Step 2. 80:20 in 10 minutes

Total run time, less equilibration time: 15 minutes

The confirmation analysis is for qualitative use only. The analyte is considered confirmed if it falls within its respective RT window on the cyano column.

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

Attachment 1

Method Detection Limits
Explosive Residues in Water Matrix

Comoound	EQL (µg/L)
HMX	0.40
RDX	0.40
1,3,5-Trinitrobenzene	0.40
1,3-Dinitrobenzene	0.40
Tetryl	0.40
Nitrobenzene	0.40
2,4,6-Trinitrotoluene	0.40
4-Amino-2,6-dinitrotoluene	0.40
2-Amino-4,6-dinitrotoluene	0.40
2,4-Dinitrotoluene	0.40
2,6-Dinitrotoluene	0.40
2-Nitrotoluene	0.40
3-Nitrotoluene	0.40
4-Nitrotoluene	0.40

NOTE: 0.40 µg/L has been determined to be the lowest reporting limit achievable keeping an appropriate signal to noise ratio on the cyano column confirmation analysis; although all calculated MDLs, with the exception of Tetryl, are considerably lower.

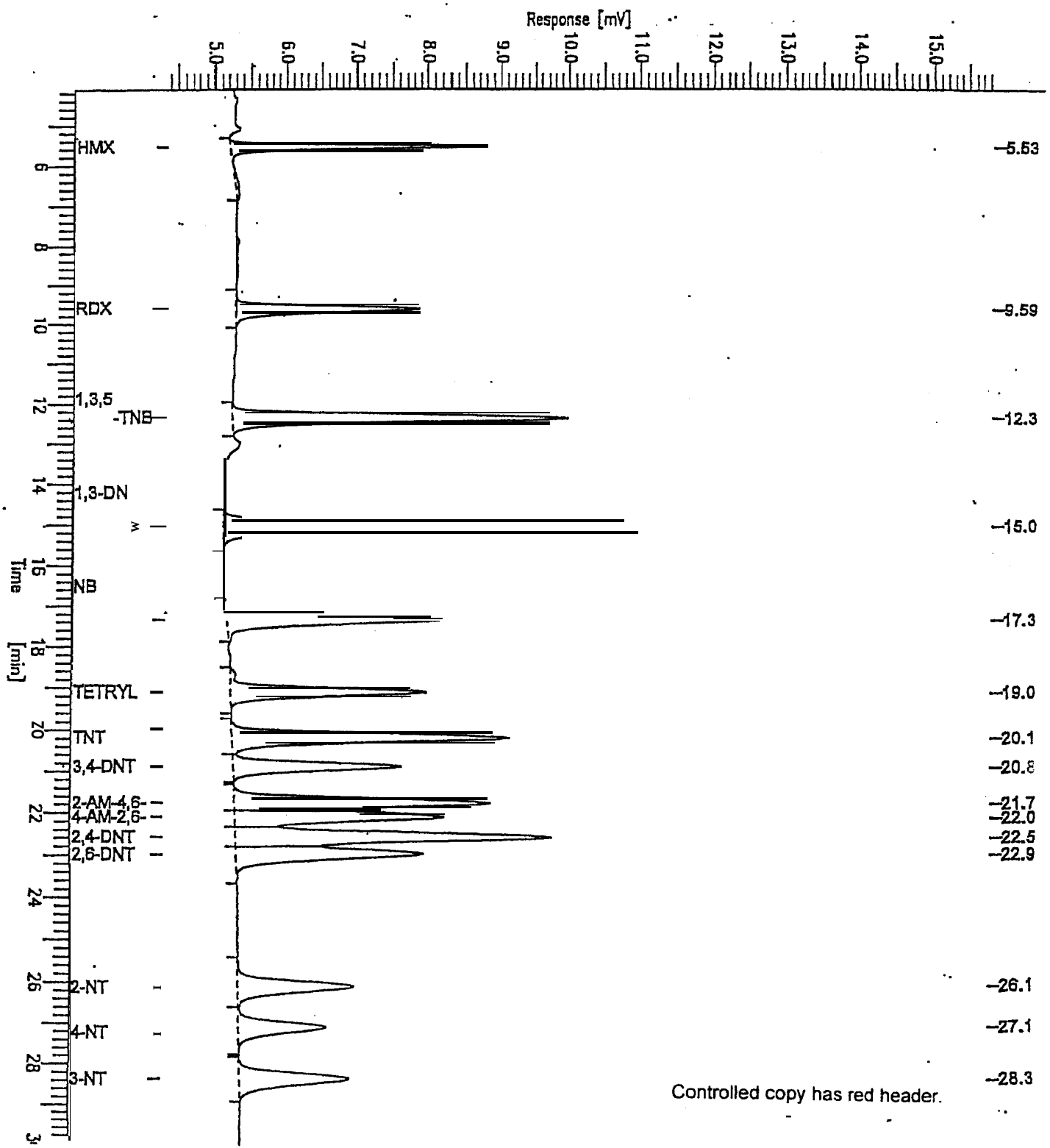
Attachment 2

Precision and Accuracy
Explosive Residues
Water Matrix

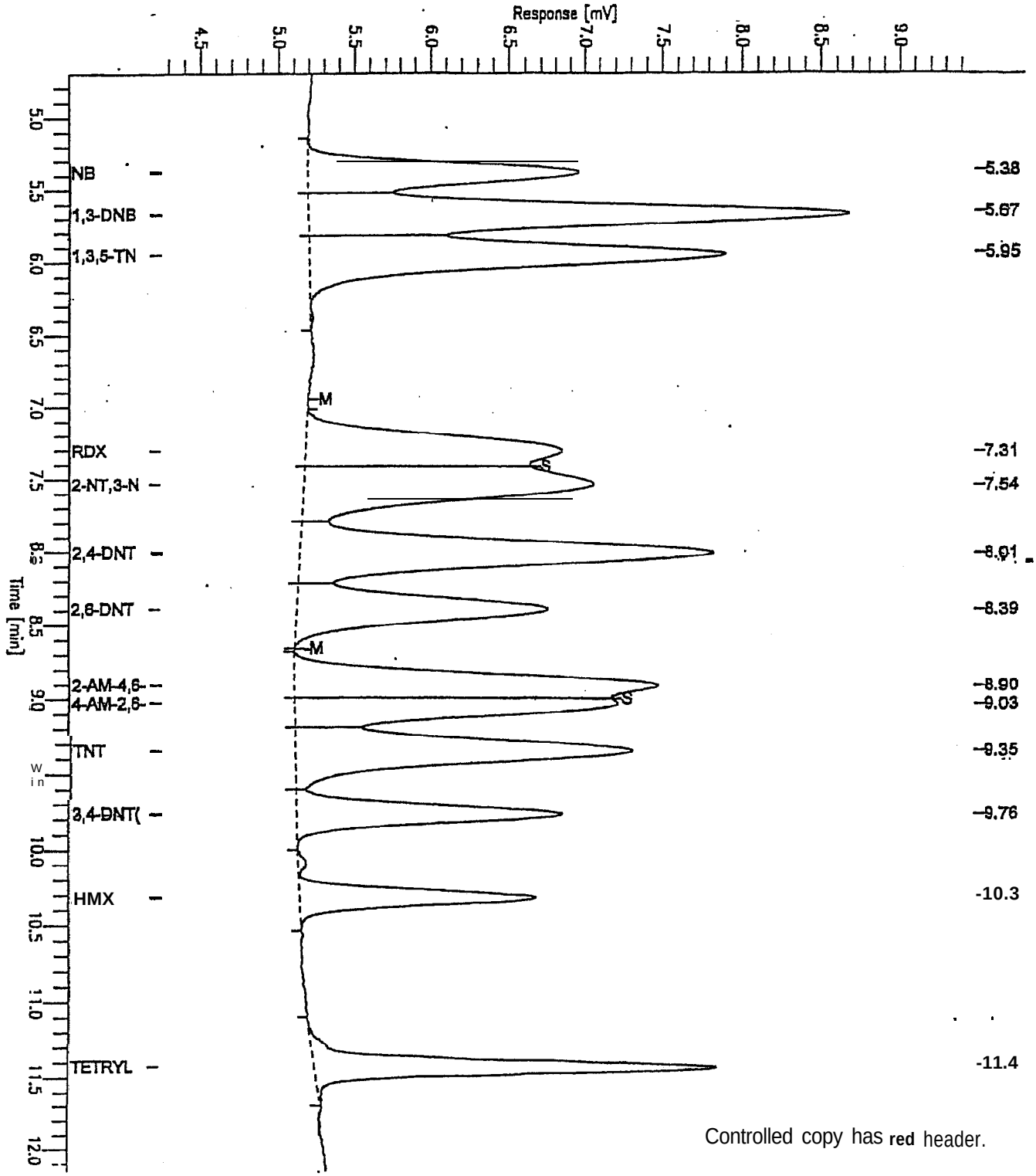
Compound	Matrix Spike Limits (%)	Control Spike Limits (%)
HMX	50 - 140	60-120
RDX	50 - 140	60-120
1,3,5-Trinitrobenzene	50 - 140	60-120
1,3-Dinitrobenzene	50 - 140	60-120
Tetryl	45 - 145	45 - 145
Nitrobenzene	50 - 140	60 - 120
2,4,6-Trinitrotoluene	50 - 140	60-120
4-Amino-2,6-DNT	50 - 140	60-120
2-Amino-4,6-DNT	50 - 140	60 - 120
2,4-Dinitrotoluene	50 - 140	60 - 120
2,6-Dinitrotoluene	50 - 140	60 - 120
2-Nitrotoluene	50 - 140	'60-120
3-Nitrotoluene	50 - 140	60 - 120
4-Nitrotoluene	50 - 140	60 - 120
3,4-DNT (surrogate)	60 - 140	60 - 140

Advisory control limits are to be used until a sufficient number of points are generated to calculate in-house limits. The limits in use may differ from those listed.

Attachment 3
Example Chromatogram
Alltech BDS C18 Column



Attachment 4
Example Chromatogram
Phenomenex Luna CN



EN CHEM MADISON LABORATORY ANALYTICAL METHODS, QA OBJECTIVES FOR AQUEOUS SAMPLES Effective June 2001 to June 2002									5/4/2001		
Parameter	units	Method (prep)	Method (analytical)	LCS (% Rec.)	LCS (% RPD)	Accuracy (% Rec.)	Precision (% RPD)	MDL	Reporting Limit	W. Length Alt. Isotope	En Chem SOP #
HPLC 8330											
1,3 - Dinitrobenzene	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0648	0.40	N/A	SVO-71
1,3,5 - Trinitrobenzene	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0605	0.40	N/A	SVO-71
2 - Amino - 4,6 - dinitrotoluene	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0749	0.40	N/A	SVO-71
2 - Nitrotoluene	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0564	0.40	N/A	SVO-71
2,4 - Dinitrotoluene	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0608	0.40	N/A	SVO-71
2,4,6 - Trinitrotoluene	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0700	0.40	N/A	SVO-71
2,6 - Dinitrotoluene	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0449	0.40	N/A	SVO-71
3 - Nitrotoluene	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0719	0.40	N/A	SVO-71
4 - Amino - 2,6 - dinitrotoluene	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0550	0.40	N/A	SVO-71
4 - Nitrotoluene	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0586	0.40	N/A	SVO-71
HMX	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0507	0.40	N/A	svo-71
Nitrobenzene	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0781	0.40	N/A	SVO-71
RDX	ug/L	8330	8330	60-120	N/A	50-140	0-50	0.0807	0.40	N/A	SVO-71
Tetryl	ug/L	8330	8330	45-145	N/A	45-145	0-60	0.0908	0.40	N/A	SVO-71

Effective June 2001 to June 2002

 "*" - SOP not available at time of release.Contact your En Chem Represenative for further assistance.

APPENDIX C

ANALYTICAL METHOD SOP DOCUMENTS

En Chem, Inc.
Madison Laboratory
Quality Assurance Manual.

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www.enchem.com

QUALITY MANUAL
FOR
EN CHEM, INC. - MADISON, WI

REVISION 1

Prepared by:
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EFFECTIVE DATE:
June 1, 2001

David Turriff, Laboratory Director — 5-30-01 Date

Gregory J. Graf, QA Officer — 5-30-01 Date

Daniel M. Rude, Technical Director — 5-29-01 Date

Glen A. Coder, Operations Manager — 5-29-01 Date

COPY NUMBER: _____

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1.0 POLICY STATEMENT

This Quality Manual summarizes the policies and procedures associated with En Chem, Inc. (En Chem) laboratory, located in Madison, WI. Specific protocols for sample handling and storage, chain-of-custody, laboratory analyses, data reduction, corrective action, and reporting are described. Adherence to procedures listed in this manual shall be the responsibility of all En Chem employees of the Madison Laboratory. Laboratory management shall be responsible for seeing that procedures and practices described in this'manual, and all referenced Standard Operating Procedures (SOPs), are fully implemented. In the event that a signatory leaves the company, for any reason, the documents shall remain in effect, to allow for revision, for a maximum period of one year or, until the document is archived. Other signatories, as well as the replacement for the vacated position, shall be responsible for implementation of the practices and procedures described in the laboratory documents.

The En Chem Madison Laboratory performs chemical analyses for inorganic and organic constituents in water, soil, and biological matrices.

The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. This includes a review of facilities and instrumentation, staffing, and any special QC or reporting requirements. All measurements are made using published reference methods or methods developed by En Chem Madison. All methods are validated according to the procedure described in Appendix B prior to use.

Any unusual requests, such as lower detection limits or additional QC, that are specified on the work order or project QAPP, will take precedence over this QA Manual if they conflict.

This Quality Manual shall be reviewed at least annually and updated as required.

1.1 Mission Statement

En Chem exists to provide innovative ideas, services, and tools to foster the revitalization of the environment.

1.2 National Environmental Laboratory Accreditation Conference (NELAC) Compliance

All policies and procedures have been structured in accordance with the NELAC standards adopted in July 1998 and applicable EPA requirements, regulations, guidance, and technical standards. This manual has been prepared in accordance with the guidance documents listed in Section 15 of this manual. Further details on these policies and procedures are contained in SOPs and related documents.

A Certification statement that addresses continual compliance with NELAC standards is included in Appendix A of this manual.

1.3 Staff Freedom From Undue Pressures

En Chem laboratory staff shall not be subject to any commercial, financial, or other undue pressures which might adversely affect the quality of their work. Any member of the staff who feels the quality of their work is potentially compromised by these, or any other influence, should bring their concerns to the attention of the QA Officer and/or the President of En Chem.

1.4 Legal identification

The Federal Taxpayer ID for En Chem is: 39-1695955. The US-EPA Laboratory ID for the Madison facility is: WI-00051,

2.0 ORGANIZATION AND RESPONSIBILITIES

2.1 Organization Chart

A corporate organizational chart for En Chem is shown in Appendix C, Figure 1. This chart includes all En Chem Laboratories and Service Offices. The street addresses and phone numbers for each location are listed in Figure 2.

An organization chart for the Madison facility is shown in Appendix C, Figure 2. A listing of credentials for all laboratory staff is presented in Appendix D.

2.2 Laboratory Management Director

The Laboratory Director is responsible for:

- Defining the minimal level of experience and skills necessary for all positions in the laboratory. In addition to education and/or experience, basic laboratory skills are considered.
- Ensuring that all technical laboratory staff have demonstrated initial and ongoing proficiency in the activities for which they are responsible.
- Ensuring that the training of personnel is kept up-to-date.
- Documenting all analytical and operational activities.
- Supervising all personnel.
- Ensuring that all sample acceptance criteria, as defined in SOP GEN-30, are verified and that samples are logged into the sample tracking system and properly labeled and stored.
- Performing an annual Management System Review.
- Documenting the quality of analytical results reported by the laboratory.
- Ensuring that the laboratory has the appropriate resources and facilities to perform requested work.
- Ensuring that corrective actions relating to findings from the internal audit are completed.
- Nominating deputies when a Technical Director or the Quality Assurance (QA) Officer is absent.
- Responsible for overall operation of the organization including fiscal resources and personnel. Examples of these duties include business development, approval of capital investments, coordination of branch offices and long range planning.

2.3 Quality Assurance Officer

As shown in Figure 2 Appendix C, the QA Officer is independent of direct job involvement and day-to-day operations, and reports directly the Laboratory Director, to resolve any dispute involving data quality. He is responsible for the implementation of the Quality System. The QA Officer is authorized to stop work as deemed necessary in the event of serious QA/Quality Control (QC) issues. Specific functions and duties include:

- Performing QA audits on various phases of laboratory operations.
- Reviewing and approving QA plans and procedures.
- Providing QA technical assistance to project staff.
- Reporting on the adequacy, status, and effectiveness of the Quality System regularly to the Technical Director and Laboratory Director.
- Overseeing laboratory QA and QC.
- Overseeing QA/QC documentation.
- Overseeing implementation, and monitoring of, laboratory corrective actions.
- Overseeing preparation and maintenance of SOPs.
- Approval of SOPs and QA Procedures.
- Approval of any/all Quality Assurance Project Plan (QAPP).
- Serving as the focal point for QA/QC and being responsible for the oversight and/or review of quality control data.
- Having documented training and/or experience in QA/QC procedures and being knowledgeable in the quality system as defined under NELAC.
- Having a general knowledge of the analytical test methods for which data review is performed.
- Conducting internal audits on the entire technical operation annually.
- Notifying laboratory management of deficiencies in the quality system and monitoring corrective action.

2.4 Technical Directors for Chemical Analyses

The Technical Director for Chemical Analyses reports to the Laboratory Operations Manager and is responsible for:

- Coordinating laboratory analyses.
- Supervising in-house sample management.
- Scheduling sample analyses.
- Overseeing data review.

- Overseeing preparation and approval of final labortaory reports.
- Participating in the annual Management System Review.
- Certifying that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited.
- Approval of the purchase of consumables and supplies used in the laboratory.

2.5 Support Systems Supervisors

2.5.1 The Sample Receiving Group Leader shall be responsible for the following:

- Supervision and training of sample receiving staff.
- **Overseeing** the processing of daily sample receipts.
- Coordination of sample tracking and disposal within the laboratory.
- Communication of nonconformances in the receipt of samples to the project manager.

2.5.2 The Information Systems Group Leader shall be responsible for the following:

- Supervision and training of laboratory IS staff.
- Administration and maintainence of the laboratory information management system (LIMS) and the laboratory network.
- Troubleshooting and resolution of computer related problems.
- Planning and implementation of policy and changes to the laboratory information systems.

2.5.3 The Client Services Group Leader shall be responsible for the following:

- Supervision and training of the laboratory Project Managers and Project Coordinators
- Management of projects within the laboratory.

2.6 Technical Staff

Technical staff are responsible for sample analysis and identification of corrective actions. All personnel are responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function. As documented in the employee records, each technical staff member has the experience and education to adequately demonstrate knowledge of their particular function and a general knowledge of laboratory operations, analytical test methods, quality

assurance/quality control procedures and records management. A listing of all staff, positions, educational background and experience is included in Appendix D.

2.7 Training

All employees are required to read, understand, and use the latest version of each laboratory SOP, which relates to their job responsibilities. The procedures in SOP GEN-8 document the use of current SOPs. Analysts and Technicians demonstrate continued proficiency by repeating the Initial Demonstration or, acceptable performance of a blind QC Check Sample (single blind to the analyst) at least once per year. Documentation of proficiency is maintained in laboratory training files by the QA Officer.

2.8 Laboratory Capabilities

The En Chem Madison laboratory performs analyses on water, soil, and biological matrices for environmental contaminants. Analyses performed are listed in Appendix E, along with the analytical technique, literature references, and the corresponding En Chem SOP.

3.0 QUALITY ASSURANCE OBJECTIVES

The overall QA objectives of the En Chem Madison laboratory are to develop and implement SOPs for chain-of-custody, laboratory analysis, and results reporting which will provide results of known and documented quality. The procedures provide a comprehensive and effective quality control program to measure and validate laboratory performance. The system provides for the maintenance of records relating to sample submittal and the production of accurate, precise, and complete laboratory data, using approved or proven methods. In addition, the system identifies factors, which may adversely affect quality and provides for corrective action when necessary.

Several indicators are used as qualitative and quantitative descriptors in interpreting the degree of acceptability or utility of data. The principal indicators are precision, bias (accuracy), representativeness, comparability, completeness, and detection limits. These indicators, defined below in detail, provide goals for the quality of data generated in the analytical measurement process.

3.1 Precision

Precision is a measure of the degree to which two or more measurements are in agreement.

Precision is assessed through the calculation of Relative Percent Difference (RPD) between a sample matrix spike (MS) and matrix spike duplicate (MSD). The RPD is compared to acceptance limits derived from historical laboratory data or from control limits presented in a project specific QAPP. In cases where an insufficient quantity of sample is available for matrix spikes the precision will be evaluated by calculation of the RPD between a Laboratory Control Sample (LCS) and a LCS Duplicate (LCSD).

The RPD is also determined through the assay of field or laboratory duplicates.

For replicate results D_1 and D_2, the RPD shall be calculated:

$$RPD = \frac{[D_1 - D_2]}{\left[\frac{(D_1 + D_2)}{2}\right]} \times 100$$

3.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference or true value. Accuracy is expressed in terms of percent recovery (%R).

$$\%R = \frac{O_i - O_s}{T_i} \times 100$$

Where:

%R = Percent Recovery

O_i = Concentration of analyte observed in the spiked sample.

O_s = Concentration of analyte observed in the unspiked sampte.

T_i = Concentration of the Spike

Accuracy is assessed through the analysis of MS/MSD, quality control check samples, laboratory control samples, and surrogate compound spikes. The % Recovery obtained is compared to control limits derived from historical laboratory data or from control limits presented in a project specific QAPP.

3.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition within a defined spatial and/or temporal boundary.

Representativeness is ensured by using the proper analytical procedures, appropriate methods and meeting sample holding times.

3.4 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected under normal conditions.

Laboratory completeness is a determination of the amount of valid measurements obtained compared to all the measurements taken in the project. The laboratory completeness objective is to generate valid data for all samples at a rate greater than 95 percent of all samples analyzed.

3.5 Comparability

Comparability is an expression of the confidence with which **one** data set can be compared to another.

Comparability is achieved by following Standard Operating Procedure, analysis within holding times, the use of approved analytical methods, use of consistent detection levels, and consistent rules for reporting data (including reporting results in common units).

3.6 Data Reporting Limits

3.6.1 Method Detection Limits

Method Detection Limits (MDLs) shall be determined for each analyte and matrix as specified in laboratory SOP LAB-14. The laboratory SOP is based on EPA guidance given in Title 40, Code of Federal Regulations, Part 136, Appendix B. Laboratory MDL determinations shall be performed during initial validation of a method, whenever there is a significant change to the test method or instrument type. MDS's shall be performed as necessary but no less than once per year. See SOP LAB-14 for more specific guidance.

3.6.2 Limit of Quantitation

The Limit of Quantitation (LOQ) is nominally 3.18 times the MDL.

3.6.3 Estimated Quantitation Limit

The Estimated Quantitation Limit (EQL) is a nominally chosen reporting limit, which is greater than the MDL. The EQL is the minimum concentration of an analyte that can be identified and quantified within specified limits of precision and bias during routine analytical operating conditions. EQLs may be adjusted to meet specific client project reporting limits. When this occurs, the value is referred to as a Project Specific Reporting Limit (PSRL).

4.0 SAMPLING PROCEDURES

The En Chem-Madison laboratory does not provide field-sampling services.

4.1 Ice Chests and Shipping Containers

Shipping containers are washed and inspected prior to and following use. Containers are rinsed with tap water and air dried before storage. If a container becomes severely contaminated or damaged, it is cleaned as thoroughly as possible, rendered unusable, and properly disposed.

4.2 Sample Containers

The laboratory provides sample containers. All sample containers are purchased from commercial sources and are precleaned and, certified by the vendor. The containers meet or exceed the requirements of "EPA Publication #9240.0-05A" Specifications and Guidance for Contaminant-Free Containers. The sample containers used for a specific project are traceable by lot number to certification statements provided by the manufacturer. These certificates are maintained on file in the laboratory.

4.3 Sample Preservation

Sample preservatives are added to sample containers in the laboratory prior to shipment to the field. Sample preservation requirements are detailed in SOP REC-3. The preparation of preservatives is documented and is traceable to the lot numbers of reagents used to prepare the preservative. The documentation procedure for laboratory reagents and solutions is detailed in SOP LAB-22, Traceability of Laboratory Reagents.

Proper preservation is the ultimate responsibility of the sampling team. The laboratory may provide additional preservatives for any site with a history indicating that non-routine preservation is required. This information should be communicated to the project manager during project start-up. The laboratory verifies proper sample preservation at the time of sample receipt. The adequacy of sample preservation shall be verified and recorded at the time of sample receipt. The preservation of samples for volatile organics is not checked until the analysis is completed.

Specific project requirements, such as notification prior to pH adjustment, shall take precedent over the laboratory SOP if these requirements have been communicated to the laboratory prior to sample receipt.

5.0 SAMPLE CONTROL AND PROCESSING

Prior to analysis, sample collection, preservation, and storage must be performed properly to maintain sample integrity. En Chem's laboratories are access-controlled facilities. Upon receipt all samples shall be inspected for leakage or breakage, and inventoried against the Chain of Custody document. The sample log-in procedure includes assignment of unique sample identification numbers, to ensure samples can be tracked, data can be stored, and quality control samples can be identified for all analyses occurring in the laboratory. Documentation of storage and laboratory handling is documented at this time. Specific procedures are summarized below, and detailed in laboratory SOPS. Deviations from the SOPs must be documented in accordance with Section 13: Corrective Action, of this manual.

5.1 Bottle Request and Chain-Of-Custody Forms

A Bottle Request Form is generated by the laboratory project manager to ensure that the proper bottle types and preservatives are made available to the project sampling team. The bottle request form is submitted to the laboratory Sample Receiving group before the sampling event. Field personnel must properly complete the sample Chain-of-Custody (COC) Form and return it to the laboratory with the samples. The COC indicates the work requested for each sample point. Work requests can also be pre-arranged with the En Chem project manager. Example Bottle Request and COC forms can be found in Appendix I.

5.2 Chain-Of-Custody Procedures

Sample custody documentation includes records necessary to trace a sample from point of origin through final report. Sample custody documentation requires the recording of each event **or** procedure to which the sample is subjected. This includes but is not limited to: field activities such as sample collection and preservation, as well as laboratory activities such as sample receipt and sample login. The COC remains with the samples during transport, and serves as a written record of sample possession and transference. A sample is considered to be in custody if it is in one's possession, is locked or sealed during shipment, or is placed in a secure area limited to authorized personnel. The COC must be signed and dated by everyone who takes possession of the samples. Samples and documents shipped by commercial carrier must be sealed in watertight containers. Shipping containers must be sealed before delivery to commercial carriers. The waybill of the carrier shall serve as an extension of the COC

between the field and the laboratory. Upon arrival at the laboratory, the shipping containers are opened in the sample log-in area. The contents are checked against the COC and any discrepancies are noted. Additions or changes to the COC are signed and dated at the time that they are made. If the discrepancies cannot be resolved. project personnel shall be notified and the samples will be held until the problem is resolved. The laboratory will not be responsible for meeting holding times on these types of problem samples. Actions taken to resolve problems with incoming samples are documented (see Section 13, Corrective Action).

Sample custody at the laboratory, is documented on the Laboratory Tracking Sheet, which is completed by the Sample Receiving Group for each batch of samples received. The use of this laboratory form is discussed in SOP REC-7.

5.3 Sample Receipt

The laboratory has a specifically designated area for sample receipt. Samples are received during normal business hours, on Saturday mornings and at other times by special arrangements. Sample receiving procedures are defined in SOPs. These procedures include completion of a cooler receipt log (SOP REC-5), and sample log-in (SOP REC-7). Refer to the specific laboratory SOP for further detail.

5.3.1 Sample Acceptance Policy

En Chem's written sample acceptance policy, SOP GEN-30, requires the following:

- Proper, full, and complete documentation-, including the sample identification, the location, date, and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;
- Unique identification of samples using durable labels completed in indelible ink;
- Use of appropriate sample containers.;
- Receipt within holding times;
- Adequate sample volume; and
- Procedures to be used when samples show signs of damage or contamination.

Samples, which do not meet the acceptance criteria, must be documented on a Sample Entry Nonconformance Memo (SOP GEN-15). These memos are routed to the En Chem Project Manager of the project for resolution with client project staff. Analytical

results from non-acceptable samples shall be qualified or otherwise explained in the laboratory report.

5.4 Sample Storage

The primary considerations for sample storage are proper temperature as specified by method requirements and the completion of extraction and analysis within the specified holding times. Sample receiving personnel are responsible for ensuring that samples are initially property stored.

Samples stored in a particular refrigerator, freezer, or other storage area shall be recorded in a logbook for that storage area. Documentation of sample storage is provided on the Laboratory Tracking Sheet (See Appendix I), which must be completed for each sample batch.

To minimize the possibility of contamination all samples for volatile organics are segregated in a refrigerator specifically designated for these samples. A second volatiles refrigerator **is** used to segregate known high level samples, or those with a noticeable odor at the time of receipt.

All samples, sample fractions, extracts, lechates, or other sample preparations shall be stored according to the conditions listed in the authoritative test method. These conditions are detailed in the specific method SOP's.

5.5 Sample Disposal

Samples may be completely consumed during analysis, returned to the client or sampling location, or stored under appropriate environmental conditions if re-analysis is anticipated. Ambient storage is used if re-analysis is not likely. Samples and extracts are disposed of not earlier than thirty days after issuance of the final report unless otherwise specified. Samples are placed into long-term storage following analysis, or returned to the client if required by the project.

The En Chem Madison facility is classified as a small quantity waste generator by the USEPA. Disposal of all samples, hazardous and nonhazardous, is performed in accordance with all applicable local, state, and federal environmental regulations. Some nonhazardous wastes may be disposed of in a sanitary sewer as permitted by 40 CFR 261.3 (a),(2),(iv). Hazardous wastes as defined under 40 CFR 261 are stored in designated locations in the laboratory according to EPA standards.

En Chem has an agreement with a licensed hazardous waste shipper to pack, test, and ship the hazardous waste approximately every four weeks. Hazardous wastes are shipped to licensed waste disposal facilities for disposal., En Chem receives a Certificate of Disposal for all disposed material. All documents related to waste management are maintained on file in the laboratory.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

Instruments and equipment used to generate data shall be calibrated with sufficient frequency, and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications. Calibration may be of two types: operational calibration which occurs before instrument use, or periodic calibration which occurs at prescribed intervals. This section describes procedures for maintaining the accuracy of all instruments and measuring equipment that are used for conducting laboratory analyses.

6.1 Traceability of Calibration

The calibration of analytical equipment and instruments shall be traceable to the National Institute of Standards and Technology (NIST).

6.2 NIST Traceable Reference Standards

Physical standards used to verify laboratory equipment in used in the labs shall be traceable to nationally recognized standards such as the NIST. The reference standards shall be administered by the Quality Assurance officer. Reference standards must be re-certified according to the expiration dates on the certificate for each standard. If no expiration date is specified a time period of three years shall be used.

Chemical reference standards that are purchased from commercial vendors shall be traceable to the NIST. Calibration certificates that accompany standard materials when received in the laboratory are maintained on file in the laboratory.

6.3 General Requirements

Each calibration shall be supported by documentation indicating calibration date, method, instrument, analyst, analysis date, analyte, concentration and response (or response factor). Sufficient information must be recorded to permit reconstruction of the calibration. Acceptance criteria for calibrations comply with method references or QAPP requirements. This documentation is referenced in, or kept with, data files or analytical log books.

NOTE: Separate records are kept for periodic calibration. These items shall be filed and archived by the laboratory QA Officer.

6.4 Analytical Support Equipment

Analytical support equipment includes: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices and volumetric dispensing devices. If quantitative results are dependent on their accuracy, as in standard preparation and dispensing or diluting procedures, then all such support equipment **is** maintained in proper working order. The records of all maintenance including service calls shall be recorded in the equipment maintenance log or filed with the QA Officer.

Calibration is verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration must be within the specifications required of the application for which is equipment is used. Noncompliant equipment is removed from service until repaired.

Any equipment which is not calibrated at least annually must be clearly labeled as 'Not Calibrated', such as an oven which is only used to dry glassware. Equipment which is not in use shall be clearly labelled as such.

6.4.1 Temperature Monitoring

Each working day, the temperatures of ovens, refrigerators, freezers, and water baths are checked with calibrated thermometers which are traceable to NIST references. The temperatuers of these units are recorded in the logbook which contains the acceptance limits for that unit. SOP's LAB-l 2 and LAB-l 3 describe these procedures and corrective action for noncompliance.

All therometers used in the laboratory shall be verified with a NIST traceable thermometer, in the range of use, prior to being used in ite laboratory and once annually thereafter. Each thermometer shall contain **a** calibration tag which identifies the thermometer with a unique number and shows the date which reverification is due. The procedure for calibration verification is available in SOP LAB-IO.

6.4.2 Balances

Laboratory balances shall be checked with Class 'S' weights traceable to NIST standards, which bracket the range of use, each day prior to use. The calibration check shall be recorded in the logbook which contains the acceptance limits for that unit. SOP LAB-26 describes this procedure and corrective action.

All balances are serviced and calibrated two times each year by an external service provider. A calibration sticker is attached to each balance which identifies the service contractor, and contains the serial number of the balance, the calibration date, and the date that recalibration is due. Records of this balance calibration shall be maintained by the QA Officer.

6.4.3 Volumetric Dispensing Devices

Mechanical volumetric dispensing devices are checked for accuracy monthly as described in laboratory SOP LAB-l.

6.5 Instrument Calibration

Analytical instrument calibration consists of measuring a standard response or preparing a standard calibration curve.

Detailed calibration procedures for specific laboratory instruments are documented in specific instrument SOPs. The SOP for each method performed in the laboratory describes the calibration procedures, their frequency, acceptance criteria, and the conditions that require recalibration. The analyst is required to perform and document the calibration procedure prior to sample analysis.

In all cases, the initial calibration shall be verified using an independently prepared calibration verification solution. Calibration records are documented on the raw data or in the logbook for each instrument. At a minimum the following information shall be recorded or referenced in the logbook: instrument identification, calibration date, analyst, calibration solutions/concentrations analyzed.

6.5.1 Limited Calibration Procedures

The use of a limited calibration procedure is allowed on a project specific basis for unusual, non-routine compound analysis. In this case, the laboratory may run one or two standards in order to establish a retention time, determine instrument response, and establish a reporting limit. The use of limited calibration must be discussed with the client by the Project Manager prior to initiation of the project. The approval to proceed with a limited calibration for a project shall be documented in the project file by the Project Manager. The use of such procedures shall be included in the final report or accompanying case narrative.

7.0 TEST METHODS AND STANDARD OPERATING PROCEDURES

En Chem maintains Standard Operating Procedures (SOPs) that accurately reflect all test methods, assessment of data integrity, corrective actions, and handling customer complaints. The primary purpose of a Standard Operating Procedure (SOP) is to provide a "How to" document which laboratory personnel can use to perform various routine laboratory operations. SOPs are prepared and used to minimize the introduction of random and systematic errors by ensuring that all personnel use the same procedure when performing a specific operation. SOPs also act as a training guide for new personnel.

Each SOP indicates the effective date, the revision number, and the appropriate signature(s).

7.1 SOP Preparation and Organization

Laboratory SOP GEN -1, Preparation of Standard Operating Procedures, defines the format, identification, and control of SOPs. All SOP's shall be reviewed annually for any needed revisions.

7.2 SOP Control and Distribution

SOPs are located throughout the laboratory are printed on paper with a red header signifying that the copy is a controlled document. The controlled copies shall be issued and distributed by the QA Officer or a designee. Each laboratory area has copies of the appropriate SOPs pertaining to that work area. The SOPs shall be accessible to all personnel in their immediate work areas.

Uncontrolled copies of SOPs may be sent to clients for inclusion in workplans, etc. These uncontrolled copies are are photocopies of the controlled SOPs that do not have the red header and are, therefore, easily identified as uncontrolled copies.

7.3 SOP Archival

When an SOP is revised, the original of the previous revision shall be archived by the QA Officer. These archived SOPs shall be retained indefinitely for future reference. The archive is maintained in a locked file cabinet for future reference.

7.4 SOP Formats for Test Methods

Procedures describing how analyses are actually performed in the laboratory. are specified in method SOPs. SOPs for sample preparation, cleanup, and analysis are based on literature references published by the US-EPA, ASTM, and other organizations and on internally developed methods validated according to EPA's Performance-Based Measurement System. Examples of items included or referenced in a Method SOP include:

1) Identification of the test method.
2) Applicable matrix or matrices.
3) Method detection limit.
4) Scope and application, including components to be analyzed.
5) Summary of the method.
6) Definitions.
7) Interferences.
8) Safety.
9) Equipment and supplies.
10) Reagents and standards.
11) Sample collection, preservation, shipment and storage.
12) Quality control.
13) Calibration and standardization.
14) Procedure.
15) Calculations.
16) Method performance.
17) Pollution prevention.
18) Data assessment and acceptance criteria for quality control measures.
19) Corrective actions for out-of-control data.
20) Contingencies for handling out-of-control or unacceptable data.
21) Waste management.
22) References.
23) Any tables, diagrams, flowcharts and validation data.

7.5 Laboratory SOP Listing

A complete listing of En Chem Madison SOPs are located in Appendix J.

7.6 Deviations From Laboratory SOP

Deviations from laboratory SOP may occur as part of a planned process such as a project work plan which specifies added or altered procedures to improve method performance in samples from that site. In this case the deviations shall be included in the case narrative for that project. Unplanned deviations from SOP's, such as an oversight by staff, shall be documented on a nonconformance memo. See Section 13, and En Chem SOP GEN -15. These deviations shall be included in the final report or accompanying case narrative.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 Laboratory Quality Control Samples

The data acquired from QC procedures are used to estimate the quality of analytical data, to determine the need for corrective action in response to identified deficiencies, and to interpret results after corrective action procedures are implemented. Each method SOP includes a QC section which addresses the minimum QC requirements for the procedure. The internal QC checks may differ slightly for each individual procedure but in general are described below. The acceptance limits and corrective actions for these QC checks are described in Section 12 and 13 of this manual.

8.1 .1 Blanks

a) Method Blank

A method blank is a blank of appropriate analyte-free matrix that **is** processed (digested, extracted, etc.) and analyzed with a specified sample set. The purpose of the method blank is to verify that interferences caused by contaminants in the solvents, reagents, glassware, etc. are known and minimized. Method blanks are performed at a frequency of one per batch of 20 samples or less, per matrix type, per sample preparation whichever is more frequent. The method blank is processed through all clean-ups, etc., which were performed on the samples in the batch. Method Blank results are used to determine batch acceptance. Acceptance criteria are presented in Section 12.4.

b) Trip Blank - Volatile Organics

Trip Blanks are routinely supplied and analyzed for volatile organics. Trip blanks are necessary because volatile organics samples are susceptible to contamination by diffusion of organic contaminants through the Teflon-faced silicon rubber septum of the sample vial. Trip Blanks **are** prepared by filling two 40-mL VOA vials with organic free water. These vials are then shipped with the field kit, and follow the sample bottles through the field collection and return shipment to the laboratory. Trip blanks are analyzed and reported in the same manner as samples.

c) Other Blanks

Other types of field quality control blanks, such as field and rinsate blanks are analyzed and reported in the same manner as samples.

8.1.2 Spiked Laboratory Control Samples

a) Laboratory Control Samples (LCS)

A laboratory control sample consists of a control matrix which has been spiked with the analytes(s) of interest or compounds representative of those analytes. Laboratory Control Samples are analyzed at **a** minimum of 1 per batch of 20 or fewer samples per matrix type per sample extraction or preparation method. Results of the LCS are expressed in terms of percent recovery, and are used to determine batch acceptance. Acceptance limits are established based on in-house data. Table1 of Section 12 provides interim limits for use prior to calculation of in-house limits.

Tests for which no spiking solutions are available, or spiking is not applicable, **a** purchased Quality Control Sample will be used in place of the LCS described above. In these cases the acceptance limits provided by the manufacturer will be used. Some examples of these tests include, total suspended solids, total dissolved solids, total volatile solids, and oil and grease.

b) Laboratory Control Sample Duplicates (LCSD)

A second LCS which is used to evaluate laboratory precision when adequate sample is not supplied by the client to perform a Matrix Spike/ Matrix Spike Duplicate. The Relative Percent Difference (RPD) will be calculated between the **LCS** and LCSD and the value evaluated against in-house control limits. The LCSD must also meet the criteria for the LCS.

8.1.3 Spiked Samples

a) Matrix Spikes (MS)

Matrix spikes are performed to evaluate the effect of the sample matrix upon analytical methodology. A separate aliquot of sample is spiked with the analyte of interest and analyzed with the sample. Matrix spikes are performed at a minimum frequency of one in 20 samples per matrix type per sample extraction or preparation method and is done more frequently where regulations require. Matrix spike recoveries are evaluated against in-house control limits. Specific corrective actions for samples recoveries outside of established control limits are provided in the method SOPs. Poor performance in a matrix spike generally indicates a problem with the sample composition, and not the laboratory analysis, and results are used to assist in data assessment.

For analytes which spiking solutions are not available, such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, or turbidity, a matrix spike is not performed. In these cases a duplicate analysis is perfromed.

b) Matrix Spike Duplicates (MSDs)

A separate sample aliquot is spiked with the analyte(s) of interest and analyzed with the associated sample and sample matrix spike. Matrix spike duplicates are performed along with matrix spikes at the same frequency. Matrix spike duplicates are are evaluated for accuracy in the same manner as matrix spikes. In addition, the Relative Percent Difference (RPD) will be calculated between the MS and MSD. If the RPD is outside of the established control limits, the sample data shall qualified and all QC data will be carefully evaluated to determine if remedial action is required.

c) Surrogate Spiking (SS)

Surrogate compounds are added to all samples, standards, and blanks for all organic chromatography test methods except when the matrix precludes its use or when a surrogate is not available. Surrogate recoveries are evaluated against in-house control limits. Specific corrective actions for samples with surrogates outside of established control limits are provided in the method SOPs. Poor surrogate recovery generally indicates a problem with the sample composition and is reported to assist in data assessment.

8.1.4 Other QC Samples

a) Duplicate Analysis

Duplicate analysis may be used to calculate the precision (relative percent difference) of an analysis in cases where the levels of analyte is sufficiently above the EQL, or a spike of the analyte is not possible, i.e. TSS. Frequency of duplicate analyses may be either one sample per similar matrix group of 10 or one sample per group of 20, depending on choice of methodology. If the RPD is outside of the established control limits, the sample data shall qualified and all QC data will be carefully evaluated to determine if remedial action is required.

b) Serial Dilution

For ICP metals analysis, a serial dilution is performed on each batch of 20 samples or less. The parent sample is diluted by a factor of 5, and the result must agree within 10% of the original sample concentration. Any results that are greater than 10% will require a flag on the sample result indicating an estimated concentration due to a chemical or physical interference.

c) Recovery Test

For the recovery test, one sample is spiked with a known amount of the analyte of interest and analyzed. The percent recovery must be within the range of 85-l 15. If the recoveries are outside of that range subsequent dilutions are performed. If recoveries remain outside of the acceptable range then all samples in the digestion group are diluted and qualified.

8.1.5 Spike Components for Organic Analysis

All reportable components are in the spike mixes. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components (such as Method 8270) or components are incompatible, a subset of the listed components are used.

8.2 Method Detection Limits

Method detection limits (MDL) are determined by 40 CFR Part 136, Appendix B. Method Detection Limits are updated as deemed necessary by the Group Leader or QA Officer, but no less than once per year.

An MDL study is not performed for any component for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, dissolved oxygen, or turbidity. For these types of analytes, the detection limit is based on a signal to noise ratio from a the analysis of QC check samples or calibration standards. The detection limit for gravimetric tests is based on the analysis of seven replicates **of** a purchased QC sample. Alternativly, the detection limit may be based on the lowest reading of the balance used to perform the analysis.

b) The method detection limit is initially determined for the compounds of interest in each method in laboratory pure reagent water or Ottawa sand. For the biological matrix, canned tuna fish is used.

c) En Chem has adopted standard Estimated Quantitation Limits (EQLs). For organic analyses this is equivalent to the lowest calibration standard **in** the calibration curve. For inorganic analyses the EQL is set at a point where acceptable precision can be obtained. Reporting Limits are a function of project planning and may be modified for a specific project. Deviations from standard reporting limits must be approved by laboratory management and communicated to laboratory staff prior to project start-up.

8.3 Selectivity (Organics analysis)

a) Absolute and relative retention times aid in the identification of components in chromatographic analyses and help evaluate the effectiveness of a column to separate constituents. Acceptance criteria for retention time windows are documented in each method SOP.

b) A confirmation on a column of dissimmilar phase is performed to verify a compound identification when positive results are detected for GC analysis. Such confirmations are performed on organic tests except when the analysis involves the use of **a** mass spectrometer.

8.4 Demonstration of Method Capability

Prior to acceptance and use of any method, satisfactory initial demonstration of method performance is required. It is the responsibility of the Groupleader of the section in which the method is performed to see that this activity is completed. This initial demonstration of method performance shall be performed each time there is a significant change in instrument type, personnel or test method. Upon completion of the initial validation a certification statement is completed for each analyst documenting that this activity has been performed. The procedure used and the certification statement is found in Appendix B.

9.0 DATA REDUCTION, REVIEW, REPORTING AND RECORDS

9.1 Data Reduction and Review

Data resulting from the analyses of samples is reduced according to protocols described in the laboratory SOPs. All information used in the calculations (e.g., raw data, calibration files, tuning records, results of standard additions, interference check results, sample response, and blank or background-correction protocols) are recorded in order to enable reconstruction of the final result at a later date. Information on the preparation of the sample (e.g., weight or volume of sample used, percent dry weight for solids, extract volume, dilution factor used) is maintained in bound logbooks in order to enable reconstruction of the final result at a later date.

All data are reviewed by a second analyst or supervisor according to laboratory procedures to ensure that calculations are correct and to detect transcription errors. The results of all quality control sample analyses are reviewed, and evaluated before data are approved for reporting. Laboratory SOP GEN-6 documents procedures for data reduction, review, validation, and reporting. Errors detected in the review process are referred to the analyst(s) for corrective action.

Spot checks are performed on computer calculations to verify program validity. Computer programs used for data reduction are validated before use and verified regularly.

9.2 Report Format and Contents

The results of each test, or series of tests, are reported in a Certificate of Analysis and include all the information necessary for the interpretation of the results.

Each report includes:
1) the title “Certificate of Analysis”.
2) name and address of laboratory, and phone number with name of contact person.
3) a unique identification number and the total number of pages, with all pages sequentially numbered.
4) name of client.
5) description and unambiguous identification of the sample(s) including the client identification code.
6) identification of results for any sample that did not meet sample acceptance requirements.
7) identification of the test method used.

8) any deviations from, additions to or exclusions from SOPs, and any conditions that may have affected the quality of results, and including the use and definitions of data qualifiers.
9) measurements, examinations and derived results, supported by tables, graphs, sketches and photographs as appropriate, and any failures identified; identification of whether data are calculated on a dry weight or wet weight basis; identification of the reporting units such as ug/l or mg/kg.
10)clear identification of ail test data provided by outside sources, such as subcontracted laboratories, clients, etc.
11) clear identification of numerical results with values below the Reporting Limit.

Exceptions to this standard approach for reporting are allowed with approval of a Technical Director and are documented with a nonconformance memo.

Material amendments to a test report after issue are made only in the form of a further document, or data transfer including the statement "Supplement to Test Report, identification number."

Clients are notified promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a report.

Test results are certified to meet all requirements of the NELAC standards, or reasons are provided if they do not.

9.2.1 Data Deliverable Levels

The results of quality control samples, instrument raw data, etc. may be reported if requested on a project specific basis. The content of these reports may range from **a** summary of quality control sample results, to a fully validated stand alone document containing all raw data and supporting documentation. These requirements should be discussed with the En Chem Project Manager. The level of QC deliverables is determined by project requirements and must be specified at the time that the samples are submitted to the laboratory. Deliverable formats are presented in SOP GEN-21.

9.3 Records

Laboratory records provide the direct evidence and support for the necessary technical interpretations, judgments, and discussions concerning laboratory results. All records shall be recorded in ink, be legible, identifiable, and retrievable, and protected against damage, deterioration, or loss. All laboratory records from time of sample receipt

through data reporting and sample disposal shall be available if requested by clients or an authorized regulatory agency or court. All records referenced in this section are retained for a minimum of ten years. The hardware and software necessary to access any data stored electronically shall also be maintained. Storage for longer periods is available and should be discussed with the En Chem project manager prior to initiation of the project. Laboratory policies for record creation and archival are included in En Chem SOP's GEN-4 and GEN-25.

Laboratory records are stored off-site under contract with Datakeep, Inc. Datakeep is an insured document and media storage provider located in Madison, Wisconsin, and offers security, temperature and humidity control of all records that are placed in their control.

9.3.1 General Laboratory Operations Records

The following records shall be maintained:

- Master Sample Log - A chronological paper or computerized record of samples is maintained. This documentation is completed by the Sample Receiving Group.

- Calibration Records & Traceability of Standards/Reagents -- The frequency, conditions, standards, and records reflecting the calibration history of a measurement system are recorded.

- Instrument Maintenance Logs - A separate log is maintained for each instrument listing all maintenance and calibration performed in-house or by outside groups. These logs are maintained during the instrument lifetime and then archived.

- Performance Evaluation Records - Copies of all PE results and any associated corrective actions are maintained by the QA Officer.

- Certification Program Records - Records are maintained of all correspondence, analytical data, agency results and certification of performance from all certification programs.

- Purchased Material Certificates - Information which verifies that purchased materials meet the requirements of the laboratory are maintained in the laboratory.

- Audit Records - Audit reports and responses for both internal and external audits are maintained by the QA Officer.

- Computer Software Verification - Separate record of the data used to verify each software package are maintained in the laboratory.

- Periodic Calibration Records - Information on periodic calibration, i.e., thermometer and weight set calibration, are maintained by the QA Officer.

- Nonconformance Records - A copy of all nonconformance reports are maintained. Completed nonconformance memos are included in the project file.

- Instrument Run Log - A list of samples run on each instrument is maintained in the logbooks designated for that purpose.

- Standard Operating Procedures - A file of current and historical laboratory SOPs with issue dates is maintained.

- Administrative Records -- The following are maintained:
  a) Personnel qualifications, experience and training records;
  b) Initial and continuing demonstration of proficiency for each analyst; and
  c) A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record.

9.3.2 Sample Specific Records

- Sample Management -- A record of all procedures to which a sample is subjected while in the possession of the laboratory is maintained. These include records pertaining to:
  a) Sample identification, receipt, acceptance or rejection and log-in;
  b) Sample preservation including appropriateness of sample container and compliance with holding time requirement;
  c) Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records;
  d) Disposal of hazardous samples including the date of sample or subsample disposal and name of the responsible person;

- Original Data -- The raw data and calculated results for all samples is maintained in laboratory notebooks, logs, benchsheets, files or other sample tracking or data entry forms. Instrument output is stored in a computer file or a hard copy report. These records include:

a) Laboratory sample ID code.
b) Date of analysis.
c) Instrumentation identification and instrument operating conditions/parameters;
d) Analysis type and sample preparation information, including sample aliquots processed, cleanup, and separation protocols.
e) All manual , automated, or statistical calculations.
f) Confirmatory analysis data, when required to be performed.
g) Review history of sample data.
f) Analyst's or operator's initials/signature,

- QC Data -- The raw data and calculated results for all QC samples and standards are maintained in the manner described in the preceding paragraph. Documentation allows correlation of sample results with associated QC data. Documentation also includes the source and lot numbers of standards for traceability. QC samples include, but are not limited to, control samples, method blanks, matrix spikes, and matrix spike duplicates.

- Correspondence -- Correspondence pertinent to a project is maintained in the project files.

- Deviations -- Records of all deviations from SOPs. Deviations are reviewed and approved by the QA Officer or Technical Director through the use of a nonconformance memo.

- Final Report -- A copy of any report issued and any supporting documentation.

9.4 Document Control System

A document control system, under the direction of the QA Officer, is used to ensure that all staff have access to current policies and procedures at all times. Documents which are managed by this system include this Quality Manual and all SOPs. The policy for laboratory document control, distribution, receipt, return, and accessability is maintained in SOP GEN-2.

All quality documents (this Manual, SOPs, policies, etc.) are reviewed and approved by the QA Officer, the Technical Directors and the Laboratory Director. A system for SOP and QAM review, revision and approval is documented in SOP GEN-8. Such documents are revised whenever the activity described changes significantly. All documents are reviewed at least every 3 years.

9.5 Confidentiality

All information related to a project, such as laboratory results, associated raw data, product information, processes, designs or strategies are kept in confidence to the customer who requested the analyses. This policy is documented in SOP GEN-23. Access to laboratory records and LIMs data is limited to laboratory personnel except with the permission of the QA Officer or Laboratory Director. NELAP-related records are made available to authorized accrediting authority personnel.

Where clients require transmission of test results by telephone, facsimile or other electronic means, staff will ensure confidentiality is preserved. Copies of all information related to specific samples that is sent to, or received from, clients shall be maintained in the project file for that batch number.

10.0 PERFORMANCE **AND SYSTEM AUDITS AND FREQUENCY**

10.1 Internal Laboratory Audits

Annual internal audits are performed by the QA Officer to verify that laboratory operations continue to comply with the requirements of the quality system. Where the audit findings cast doubt on the correctness or validity of the laboratory's results, an immediate corrective action is initiated and any client whose work may have been affected is notified.

The internal system audits include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc. Internal audits are conducted according to the procedures and schedule included in SOP QAU-1.

10.2 Performance Audits

Proficiency test samples are analyzed two times per year from a NIST-approved PT provider for all analytes and matrices, as applicable. Additional samples, such as make-up samples to demonstrate corrective action, may be ordered from another approved PT provider at the discretion of the QA Officer.

In addition, the laboratory performs the following QC practices to monitor the quality of the laboratories analytical activities:

a) A minimum of three rounds of internal performance evaluation samples which are purchased from an outside vendor. Additional full or partial rounds may be analyzed at the discretion of the QA Officer.

b) Use of certified reference materials where applicable.

10.3 Managerial Review

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

corrective actions and other relevant factors. Documentation of the Management Review Meeting is maintained on file by the QA Officer.

11.0 FACILITIES, EQUIPMENT, REAGENTS, AND PREVENTATIVE MAINTENANCE

11.1 Equipment and Reference Materials

'A listing of laboratory instrumentation is provided in Appendix F.

Records are maintained for all major equipment and all reference materials significant to the tests performed. These records include documentation on all routine and non-routine maintenance activities and reference material verifications.

The records include:
1) the name of the equipment;
2) the manufacturer's name, model identification, and serial number or other unique identification;
3) date received and date placed in service (if available);
4) current location, where appropriate;
5) if available, condition when received (e.g. new, used, reconditioned);
6) copy of the manufacturer's instructions, where available;
7) dates and results of calibrations;
8) details of maintenance carried out to date and planned for the future; and
9) history of any damage, malfunction, modification or repair.

11.1.1 Glassware Cleaning

Glassware is cleaned to meet the sensitivity of the method. Laboratory SOPs are available for cleaning glassware for each type of analysis performed in the laboratory, i.e. metals, semivolatile organics, volatile organics, wet chemistry. The SOP for each type of glassware is posted in the immediate area in which the glassware is cleaned.

11.2 Documentation and Labeling of Standards and Reagents

Records are kept for all standards, including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if supplied), the date of receipt, recommended storage conditions, and an expiration date. Standards, which have aged beyond the stated expiration date, must be clearly labeled as expired and cannot be used for reportable analyses. Bound logbooks are maintained to document reagent and standard preparation. These records indicate traceability to purchased stocks or neat compounds, a description of reference to the method of preparation, date of preparation, expiration date and preparer's initials. This requirement is documented in SOP LAB-22.

Original containers provided by the vendor are labeled with an expiration date if one does not exist.

All containers of prepared reagents and standards bear a unique identifier and expiration date and are linked to the documentation requirements above.

1) Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents must meet the minimum purity requirements specified by the method. For items that are not routinely ordered, the labels on the containers are checked to verify that the purity of the reagents meets the requirements of the particular method. Manufacturers lot numbers of all solvents and reagents are recorded in preparation logbooks

2) Water - The quality of reagent water sources is monitored and documented to meet method specified requirements. The specific tests performed to verify reagent water acceptability are documented in laboratory SOP LAB-l 5.

11.3 Computers and Electronic Data Related Requirements

Where computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data:

- Computer software is documented to be adequate for use.
- Procedures are established and implemented for protecting the integrity of data.
- Computer and automated equipment are maintained to ensure proper functioning.
- Appropriate procedures are used for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records. This includes the use of passwords and security levels for all staff. The security of the computer systems is the responsibility of the system administrator.

11.4 Preventative Maintenance

Preventive maintenance is performed to ensure proper instrument and equipment performance and to minimize the occurrence of instrument and equipment failure during use. Factors considered when scheduling or performing preventive maintenance include: instrument type, equipment and parts that are subject to wear, deterioration or other changes in operational characteristics, spare parts that should be available to minimize downtime, and the frequency that maintenance **is** required. Maintenance must be performed when instrument performance begins to deteriorate as made evident by calibration failure, loss of sensitivity, or failure to meet quality control criteria.

Major equipment in the laboratory may either be covered under manufacturer service contacts or trained En Chem staff. Manufacturer service technicians or En Chem staff performs periodic preventive maintenance. The analyst responsible for the instrument performs daily or routine preventive maintenance. Group leaders and section supervisors will monitor this activity. An adequate supply of consumable parts and hardware will be maintained to ensure continued instrument operation.

11.4.1 Documentation of Preventative Maintenance

Each instrument will have a maintenance log that is kept by the instrument. All maintenance must be documented; this includes maintenance performed by instrument manufacturer, and service technicians, as well as routine maintenance performed by the analyst. The analyst shall be responsible for maintaining documentation. The record of maintenance will note any parts replaced as well as observations made.

11.5 Inspection/Acceptance Requirements for Supplies and Consumables

Labels indicating the following information on receipt and testing shall be used for critical supplies and consumables.

- Unique identification number (if not clearly shown).
- Date received.
- Date opened.
- Expiration date.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO EVALUATE DATA QUALITY

Quality control acceptance criteria are used to determine the validity of the data based on the analysis of internal quality control check (QC) samples (see Section 8.0). The specific QC samples and acceptance criteria are found in the laboratory Quality Control literature. Typically, acceptance criteria are based on published EPA methods for analysis where there is insufficient data to generate limits.

Acceptance criteria for bias are based on the historical mean recovery plus or minus three standard deviation units, and acceptance criteria for precision range from zero (no difference between duplicate control samples) to the historical mean relative percent difference plus three standard deviation units.

Analytical data generated with QC samples that fall within prescribed acceptance criteria indicate the laboratory was in control. Data generated with QC samples that fall outside the established acceptance criteria indicate the laboratory was "out-of-control" for the failing tests. These data are considered suspect and the corresponding samples are reanalyzed or reported with qualifiers.

Many published EPA methods do not contain recommended acceptance criteria for QC sample results. Where no criteria exist, the laboratory uses acceptance criteria established by management policy. In these situations, En Chem uses the following as interim acceptance criteria for recoveries of spiked analytes until in-house limits are developed :

Analysis	LCS/LCSD Targets % Recovery	LCS/LCSD Targets %RPD	MS/MSD Targets % Recovery	MS/MSD Targets %RPD
Metals	80-120	20	75-125 [1]	20
Wet Chemistry	80-120	20	75-125	20
Volatile Organics	70-130	40	70-130	40
Volatile Gases	50-150	50	50-150	50
Base/neutrals	50-130	40	50-150	40
Acids	20-140	40	20-120	40
Herbicides	40-160	40	40-160	40
Organochlorine Pesticides/PCB's	50-140	40	50-140	40
Organophosphorus/Nitrogen pest.	50-150	40	50-150	40
Carbamate pesticides	70-130	40	70-130	40

The above limits apply to all matricies.
1 Also used for Biota LCS.

12.1 Laboratory Control Samples

A laboratory control sample (LCS) is analyzed with each batch of samples to verify that the accuracy of the analytical process is within the expected performance of the method. The results of the laboratory control sample are compared to acceptance criteria to determine usability of the data. Data generated with LCS samples that fall outside the established acceptance criteria are judged to be out-of-control. These data are considered suspect and the corresponding samples are reanalyzed or reported with qualifiers.

12.2 Matrix Spikes/Matrix Spike Duplicates

Results from MS/MSD analyses are primarily designed to assess data quality in **a** given matrix, and not laboratory performance. In general, if the LCS results are within acceptance criteria, performance problems with MS/MSD results may either be related to the specific sample matrix or to an inappropriate choice of extraction, cleanup, or determinative methods. If any individual percent recovery in the matrix spike (or matrix spike duplicate) falls outside the designated acceptance criteria, En Chem will determine if the poor recovery is related to a matrix effect or **a** laboratory performance problem. A matrix effect is indicated if the LCS data are within acceptance criteria but the matrix spike data exceed the acceptance criteria.

12.3 Surrogate Recoveries

Surrogates are exclusively used in organic analyses. Surrogate recovery data from individual samples are compared to surrogate recovery acceptance criteria in the laboratory's Quality Control literature. Samples which fall outside of established control limits are reextracted/reanalyzed, if sample is available, to verify the failure is matrix related. If a matrix effect is confirmed, or reextraction/reanalysis was not possible, the sample results will be qualified.

For sample extracts which are diluted, the surrogate will not be evaluated if the dilution causes the surrogate concentration in the extract to be below the lowest point in the initial calibration. In these cases, the percent recovery will be qualified with a 'D' qualifier and no corrective action required.

12.4 Method Blanks

For a method blank to be acceptable, the concentration shall not be higher than the highest of the following:

- The method detection limit, or
- Five percent of the regulatory limit of concern for that analyte, or
- Five percent of the measured concentraion in a particular sample of interest.

Each sample in the affected batch is assessed against the above criteria to determine if the sample results are acceptable. Any sample associated with an unacceptable blank is reprocessed for analysis or, if reprocessing is not an alternative, the results are reported with appropriate data qualifying codes.

13.0 CORRECTIVE ACTION

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable performance or out of control QC results that can affect data quality. All out-of-control situations or deviations from SOP must be documented on a nonconformance memo. All En Chem employees are responsible for initiating a nonconformance memo for any situation that deviates from laboratory practice or SOP. Laboratory SOP GEN-15 explains documentation, responsibilities and filing of nonconformance memos,

Nonconformances that may occur during sample receiving review include the following:

- Incomplete/missing sample documentation.
- Unacceptable sample condition.
- Samples received after expiration of sample holding times.
- Improper sample storage.
- Any other situation that might affect data quality.

Nonconformances that may occur during laboratory analysis include the following:

- Instrument failures/problems.
- Incomplete/missing sample documentation.
- Exceeding sample holding times.
- Incorrect sample preparation.
- Wrong analysis method/procedure.
- QC data (blank, spike, duplicate, surrogates, etc.) outside acceptance limits.
- Calibration requirements not met.
- Data recording, transcription or validation errors.
- Any other situation that might affect data- quality.

The QA Officer or the Technical Director is responsible for approval of the corrective action on the nonconformance memo. The QA Officer will ensure implementation and documentation of the corrective action.

Corrective actions are performed prior to release of the data from the laboratory. If necessary, a narrative will be provided in the final laboratory report.

13.1 Resolution of Client Complaints

Where a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the quality of the laboratory's tests, the laboratory shall ensure that those areas of activity and responsibility involved are promptly audited. Records of the complaint and subsequent actions are maintained in the project file. The laboratory procedure for resolution of client complaints is documented in SOP Gen-29.

14.0 SUBCONTRACTING AND SUPPORT SERVICES AND SUPPLIES

14.1 Subcontracting Laboratory Services

En Chem clients shall be advised prior to any analyses being subcontracted to another laboratory. Any subcontracted work is placed with another NELAC accredited laboratory, where required, for the tests to be performed. Procedures for subcontracting analyses are documented in laboratory SOP REC-4. The following records of all subcontracted analyses are maintained:

- a copy of the subcontracted laboratory's scope or statement of accreditation
- a copy of the report from the subcontracted laboratory
- the notice to the client.

14.2 Outside Support Services and Supplies

En Chem, Inc. uses only those outside support services and supplies that are of adequate quality to sustain confidence in the laboratory's tests. Records of suppliers for support services or supplies required for tests are maintained.

15.0 REFERENCES

NELAC Standards, July 1998

Example NELAC Quality Manual, Catalyst Information Resources, L.L.C.; Evergreen, c o

Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80, December 29, 1980, Office of Monitoring Systems and Quality Assurance, ORD, US EPA, Washington, DC 20460.

RCRA QAPP Instructions, US EPA Region 5, Revision: April 1998

ASTM D-5283-92. Generation of Environmental Data Related to Waste Management Activities: Quality assurance and Quality Control Planning and Implementation.

"American National Standards Specification and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs (ANSI/ASQC E-4)", 1994

EPA 2185 - Good Automated Laboratory Practices, 1995

ISO/IEC Guide 25: 1990. General requirements for the competence of calibration and testing laboratories.

QA/R-2: EPA Requirements for Quality Management Plans, August, 1994

QA/G-4: Guidance for the Data Quality Objectives Process, EPA/600/R-96/055, September, 1994

QA/R-5: EPA Requirements for Quality Assurance Project Plans, Draft - November 1997

QA/G-5: Guidance on Quality Assurance Project Plans, EPA/600/R-98/018, February, 1998

QA/G-6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations, EPA/600/R-96/027, November, 1995

QA/G-9: Guidance for the Data Quality Assessment: Practical Methods for Data Analysis, EPA/600/R-96/084 , January, 1998

Manual for the Certification of Laboratories Analyzing Drinking Water, EPA/570/9-90/008

APPENDIX A

NELAC CERTIFICATION STATEMENT

APPENDIX A: CERTIFICATION STATEMENT

NELAC CERTIFICATION STATEMENT

The applicant understands and acknowledges that En Chem, Inc, is required to be continually in compliance with the National Environmental Laboratory Accreditation Conference (NELAC) standards and shall be subject to the penalty provisions provided therein.

I hereby certify that I am authorized to sign this application on behalf of the applicant/owner and that there are no misrepresentations in my answer to the questions on this application.

En Chem, Inc.
525 Science Drive
Madison, WI 53711

David Turiff Signature	David Turiff Laboratory Director/ President	5/16 Date
Gregory J. Graf Signature	Gregory J. Graf Quality Assurance Officer	5-16-2000 Date
Jeffery A. Gordon Signature	Jeffery A. Gordon Technical Director	5-16-2000 Date
Daniel M. Rude Signature	Daniel M. Rude Technical Director	5-16-2000 Date

APPENDIX B

INITIAL DEMONSTRATION OF CAPABILITY/ CERTIFICATION STATEMENT

APPENDIX B: INITIAL DEMONSTRATION OF CAPABILITY

A demonstration of capability (DOC) is made prior to using any test method, and at any time there is a significant change in instrument type, personnel or test method.

All demonstrations are documented using the form in this appendix.

The following steps are performed:

a) A quality control sample is obtained from an outside source. If not available, the QC check sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The analyte(s) are diluted in, or spiked into, a volume of clean matrix sufficient to prepare.four aliquots at a concentration approximately 10 times the method detection limit.

c) The four aliquots are prepared and analyzed according to the test method either concurrently or over a period of days.

d) Using the four results, the mean recovery x and the sample standard deviation (s) of the set (n-1) is calculated for each parameter of interest.

e) For each parameter, s and x are compared to the corresponding acceptance criteria for precision and accuracy in the method (if applicable) or laboratory-generated acceptance criteria (if there is no criteria listed in the method). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters exceeds the acceptance range, the performance is unacceptable for that parameter.

f) When one or more of the tested parameters fail at least one of the acceptance criteria, the laboratory repeats the test for all parameters that failed to meet criteria. If repeated failure occurs; locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

Demonstration of Capability
Certification Statement

Date:_____

En Chem, Inc.
525 ScienceDrive
Madison, WI, 53711

Analyst(s) Name(s)_____

Matrix: Reagent Water____ Ottowa S a n d

Method/Analyte(s) _____

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analysis of samples under the National Environmental Laboratory Accreditation Program, have met the Initial Demonstration of Capability (IDC).

2. The test method was performed by the analyst identified above.

3. A copy of the laboratory SOP is available for all personnel on site.

4. The data associated with the IDC are true, accurate, complete and self-explanatory.

5. All raw data(including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained, and, the associated information is well organized and available for review by authorized inspectors.

_____ _____

Technical Director Signature/Date

_____ _____

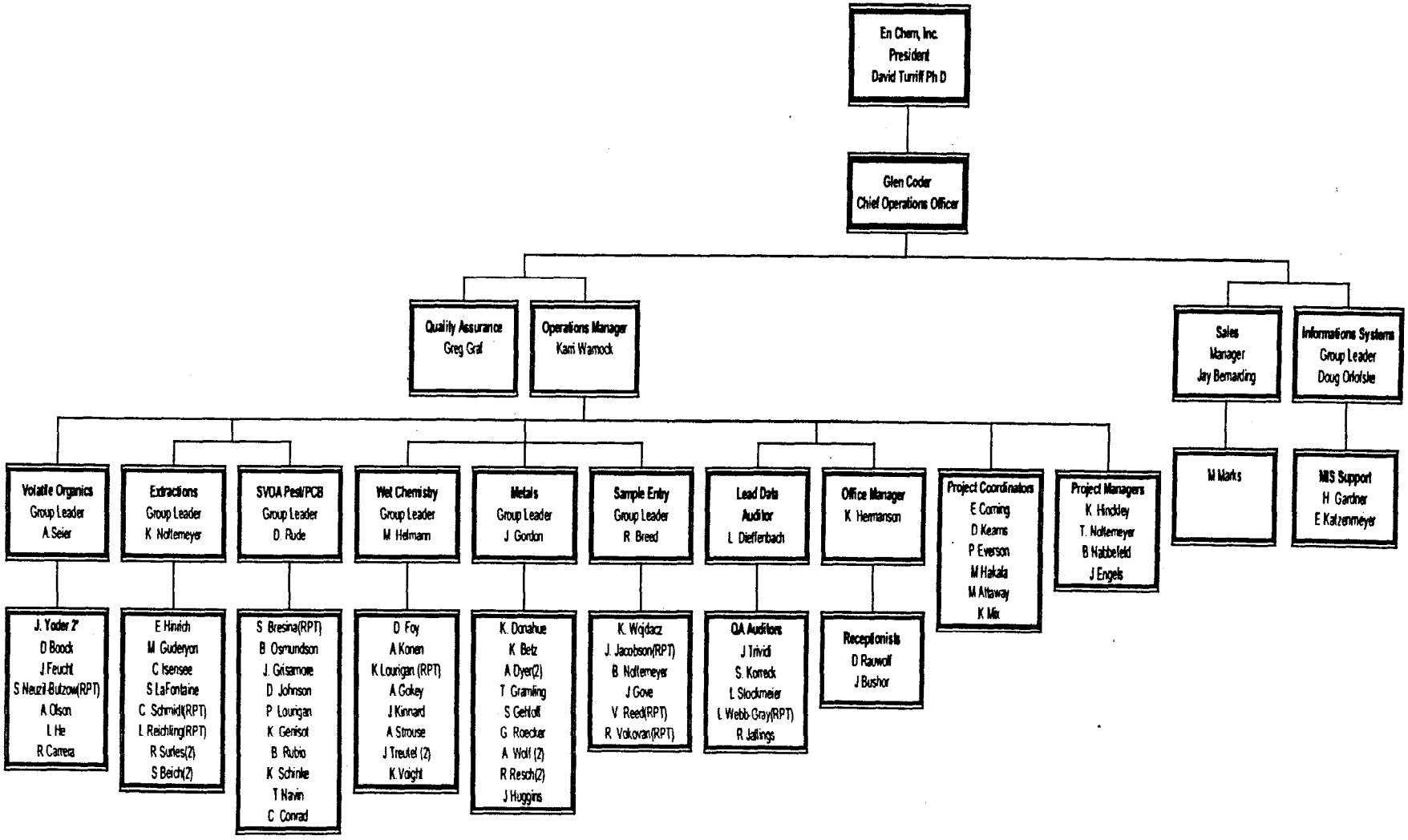
Quality Assurance Officer Signature/Date

APPENDIX C

ORGANIZATIONAL CHARTS

En Chem Organizational Reporting Structure

En Chem, Inc. Madison Laboratory



APPENDIX D

PERSONNEL SUMMARY

NAME	TITLE	DEGREE	YEARS EXP.
Glen Coder	**Operations** Manager	B.S. Communications	12
Karri **Wamock**	Client Services / Data Quality Director	B.S. Animal Science	15
Business Development I Sales			
Matt Marks	Business Development	B.S. Chemistry	29
Project Management			
Jan Engles	Project Manager	B.S. Chemistry	15
Betsy Nabbefeld	Project Manager	H.S.	9
Tod Noltemeyer	Project Manager	B.S. Biology	13
Project Coordinators			
Ellen Coming	Project Coordinator	H.S.	2
Mindy Hakala	Project Coordinator	H.S. + 2 yrs College Chem	2
Kristine Mix	Project Coordinator	B.S. Earth Sciences	1
Dawn Kerns	Laboratory Technician	B.S. Environmental Science	2
Michele Attaway	Project Coordinator	H.S.	1
Information Systems			
Doug Orlofske	MIS / LIS Technician	B.S. Geological Engineering	5
Heather Gardner	MIS/ **LIS** Technician	B.A. Journalism	4
Eli Katzenmeyer	MIS / LIS Technician	H.S. +	1
Administrative Support			
Kristine Hermanson	Administrative Assistant	H.S. +	8
Inorganic Chemistry - Metals/Wet Chemistry			
Jeff Gordon	Group Leader	B. S. Chemistry/Biology	15
Kevin Donahue	Senior Analyst	B.S. Env. Chemistry	12
Kate Betz	Senior Analyst	B.S. Biology	12
Tim Gramling	Laboratory Analyst	B.S. Chemistry	14
Gary Roecker	Laboratory Analyst	B.S. Water Chemistry	14
Doug Foy	Laboratory Technician	B.A. Biology	5
Mike Helman	Group Leader	B.S. Biology	5
Alexandria Wolf	Laboratory Analyst	B.S. Biology	2
Amanda Konen	Laboratory Technician	B.S. Biology	1
Aaron Gokey	Laboratory Technician	B.S. Biology	2
Katie Voight	Laboratory Technician	B.S. Biology	1

NAME	TITLE	DEGREE	YEARS EXP
Jason Kinnard	Laboratory Technician	B.A. Psychology	1
Sarah Gehlhoff	Laboratory Analyst	B.S. Chemistry & Environmental Science	2
Anne Strouse	Laboratory Technician	B.A. Biology	1
Jason Treutel	Laboratory Technician	B.S. Biochemistry	1
Organic Chemistry - Semivolatile			
Dan Rude	Group Leader	B.S. Chemistry	16
Denise Johnson	Laboratory Analyst	B.A. Biology	6
Kurt Schinke	Laboratory Analyst	H . S . +	6
Peggy Lourigan	Laboratory Analyst	B.S. Biochemistry B.S. Animal Science	8
Jim Grisamore	Laboratory Analyst	B.S. Biology	6
Bob Osmundson	Laboratory Analyst	B.A. Biology	8
Kevin Noltemeyer	Group Leader	H.S. +	10
Tim Navin	Laboratory Technician	B.S. Zoology, B.A.C.	2
Shelley Bresina	Laboratory Analyst	B.S. Biology	3
Bill Bruckner	Laboratory Technician	B.S.	2
Chris Conrad	Laboratory Analyst	B.S. Environmental Science	2
Betsy Hinrichs	Laboratory Technician	H.S. +	1
Chris Isensee	Laboratory Technician	B.S. Chemistry/ Biochemistry	1
Clarissa Schmidt	Laboratory Technician	B.S. Biology	1
Kristen Genisot	Laboratory Technician	B.S. Cell Biology	2
Laurie Reichling	Laboratory Technician	H.S. +	2
Mike Selby	Laboratory Technician	B.S. Anthropology/ Zoology	1
Rick Brah-Naujeck	Laboratory Technician	B.S. Geology	1
Sarah Lafontaine	Laboratory Technician	B.S. Zoology, B.A.C.	1
Organic Chemistry - Volatile			
Al Seier	Group Leader	MS. Environmental Engineering	13
Alex Olson	Laboratory Technician	H.S. +	2
Dereck Boock	Senior Analyst	B.S. Chemistry	9
Rosa Carrera	Laboratory Technician	M.S. Bacteriology	2
LiJian He	Senior Analyst	MS. Chemistry	5
Jan Feucht	Laboratory Technician	A.C. Wastewater	11
Sheila Neuzil	Laboratory Analyst	B.S. Biology	3
Jim Yoder	Second Shift coordinator	B.S. Chemical Engineering	12

NAME	TITLE	DEGREE	YEARS EXP.
Sample Receiving			
Renee Breed	Group Leader	B.S. Biology	2
Joel Gove	Laboratory Technician	B.A. Anthropology	1
Kathy Wojdacz	Laboratory Technician	B.S. Psychology	1
Bill Noltemeyer	Laboratory Technician	M.S. General Science B.E.D. Science Education	9
Quality Assurance			
Greg Graf	QA Officer	B.S. Chemistry	17
Julie Trivedi	QA Auditor	B.S. Biochemistry	8
Linda Gray	QA Auditor	B.S. Biology	12
Lynn Dieffenbach	QA Auditor	B.S. Zoology	5
Laurie Stockmeier	QA Auditor	B.S. Chemistry	4
Ruth Jallings	QA Auditor	B.S. Recreational Resource Management	6
Suzanne Korreck	QA Auditor	B.S. Microbiology	2

+ Post high school education has been or is currently being completed.

APPENDIX E

LABORATORY CAPABILITIES

APPENDIX E
LABORATORY CAPABILITIES

ANALYTE/PARAMETER	TECHNIQUE	METHOD REFERENCE		EN CHEM SOP	
Wet Chemistry					
		EPA-600	SW-846		Matrix
Acid Volatile Sulfides + SEM		Draft EPA Method		WCM-63	S
Acidity as $CaCO_3$	Titrimetric	305.1		WCM-16	w
Acidity (Free) as $CaCO_3$	Titrimetric	305.1M		WCM-57	W
Alkalinity as $CaCO_3$	LACHAT	310.2		WCM-28	W
Alkalinity. bicarb/carb as $CaCO_3$	Titrimetric	SM 23208		WCM-13	W
Ash, total	Gravimetric	160.4	- -	WCM-10	W
BOD		405.1		WCM-51	W
Bromide	I.C.	300.0	9056	WCM-50	W,S
Carbon Dioxide - Free		SM4500-CO2B			
Chloride	LACHAT	325.1	9250	WCM-19	W
(Chloride	I.C.	300.0	9056	WCM-60	W
COD (low)	Colorimetric	410.4		WCM-39	W,S
[COD (mid)	Colorimetric	410.4	----	WCM-40	W, S
Color		110.2		WCM-38	W
Conductance, specific		120.1	9050A	WCM-17	W
Cyanide, amenable		335.11335.4	90108	WCM-35	W,S
Cyanide, reactive	LACHAT	-----	7.3.3.2	WCM-23	W,S
Cvanide. total	LACHAT	335.4	9021A	WCM-23	W
Cyanide, weak and dissociable		SM 4500CN		WCM-34	W,S
Eh		SM 2580-B		WCM-56	W,S
Ferric Iron (Fe+3)	Calculated	Hach 8146160108	-----	WCM-48	w
Ferrous iron (Fe+2)		Hach 8146	- -	WCM-48	W
Flashpoint		-----	1020A	WCM-45	W,S
Fluoride - Low	I.S.E.	SM 4500F-C		WCM-42	W
Fluoride - High	I.S.E.	SM 4500F-C		WCM-42	W
Fluoride	I.S.E.	-----	MSA 26-4.3.3	WCM-43	S
Fluoride	I.C.	300.0	9056	WCM-60	W
Free liquids (paint filter)		-----	9095A	WCM-46	W,S
Hex-Chome (alk-ext/colorimetric)	Colorimetric	SM3500-CR D	3060A/7196A	WCM-61	W,S
Nitrogen, ammonia	I.S.E.	350.3	-----	WCM-49	W
Nitrogen, ammonia	LACHAT	350.1	- -	WCM-58	W,S
Nitrogen, nitrate	Calculated	353.2	-----	WCM-33	W
Nitrogen, nitrate	I.C.	300.0	9056	WCM-60	W
Nitrogen, nitrate + nitrite	LACHAT	353.2	- -	WCM-33	W
Nitrogen, nitrite	Hach	354.1		WCM-36	W,S
Nitrogen, nitrite	I.C.	300.0	9056	WCM-60	W
Nitrogen, organic	Calculated	350.1/351.4	-----	WCM-64	W,S
Nitrogen, total Kjeldahl		351.1		WCM-64	W,S

EPA-600	EPA-600/4-79-020
MSA	Method of Soil Analysis
SM	Standard Methods for the Examination of Water and Wastewater, 18th Ed.
SW-846	Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Ed.
I.C.	Ion Chromatography
I.S.E.	Ion Selective Electrode

Wet Chemistry

APPENDIX E
LABORATORY CAPABILITIES

ANALYTE/PARAMETER	TECHNIQUE	METHOD REFERENCE		EN CHEM SOP	
		EPA-600	SW-846		Matrix
Oil and Grease	Gravimetric	413.1	9071A	WCM-4	W
Oil and Grease	Gravimetric	——	9071A	WCM-41	S
pH	pH Probe	150.1	9040B/9045C	WCM-54	W,S
Phenolics, total recoverable		420.2	9066	WCM-8	W,S
Phosphate, ortho	LACHAT	365.1		WCM-44	W,S
Phosphate, ortho	I.C.	300.0	9056	WCM-60	W
Phosphorus, total		P-365.4/A-365.1		WCM-44	W,S
Solids, settleable	Gravimetric	SM2540F		WCM-55	W
Solids, total	Gravimetric	160.3		WCM-10	S
Solids, total dissolved	Gravimetric	160.1		WCM-11	W
Solids, total suspended	Gravimetric	160.2		WCM-1	W
Solids, total volatile	Gravimetric	160.4		WCM-1	W
Specific gravity		SM 2710F		WCM-47	W,S
Sulfate	LACHAT	375.2	9036	WCM-20	W,S
Sulfate, turbidimetric		375.4	SM4500-SO4E	WCM-53	W
Sulfate	I.C.	300.0	9056	WCM-60	W,S
Sulfide	Titrimetric	376.1	90308	WCM-21	W
Sulfide	Titrimetric	376.1	9030819034	WCM-22	S
Sulfide, reactive		- -	7.3.4.1	WCM-31	W
Sulfite		377.1			
Total organic carbon		- - -	9060 M	WCM-9	S
Total organic carbon		415.1	9060 M	WCM-2	W
Turbidity		180.1	- -	WCM-24	W

Total Organic Carbon as NPOC for aqueous reporting

EPA-600	EPA-60014-79-020
SM	Standard Methods for the Examination of Water and Wastewater, 18th Ed.
SW-846	Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Ed.
I.C.	Ion Chromatography

APPENDIX E
LABORATORY CAPABILITIES

METHOD REFERENCE EN CHEM SOP

Metals Prep

AQUEOUS

	EPA-600	SW-646	
Microwave Digestion		3015 / 7470A	MET-45
Acid Digestion - ICP - Total Metals	200.7	3010A	MET-3
Acid Digestion - ICP or ICPMS - Dissolved or Total Recov. Metals	-----	3005A	MET-2
Acid Digestion - Total Metals ,		3020A/7761 (Ag)	MET-4
Acid Digestion - GFAA - As and Se	-----	7060A/7740	MET-5
Hg Digestion - CVAA	245.1	7470A	MET-30

1: Analysis by GFAA or ICPMS

COMPOSITIONAL

	EPA-600	SW-846	
Microwave Digestion		3051	MET-46
Acid Digestion - ICP, ICPMS or GFAA	-----	30508	MET-6
Acid Digestion - ICP, ICPMS or GFAA - BIOTA Samples	-----	3050-Modified	MET-59
Acid Digestion - ICP, ICPMS -Ag, Sb	-----	30508 [2]	MET-7
Ha Diaestion - CVAA		7471A	MET-31

2: includes use of option 7.5 of the method.

Other Capabilities

LEACH LAB

	EPA-600	SW-846	
ASTM	- -	D3987-85	LCH-9
SPLP	- - -	1312	LCH-4
TCLP	- -	1311	LCH-2
TCLP-Neutral	- -	1311 -Modified	LCH-3
ZHE Extraction for Volatile Compounds	- -	1311, 1312	LCH-7

Metals Analysis

APPENDIX E
LABORATORY CAPABILITIES

	METHOD REFERENCE	EN CHEM SOP
	EPA-600 / SW-846	
ICP - Trace	6010	MET-42
ICP - AES	601 0B	MET-27
ICP - MS	200.8 / 6020	MET-58
GFAA - 51002 Including: Antimony	7041	MET-l 5
Arsenic	7060A	MET-21
Cadmium	7131A	MET-20
Chromium	7191	MET-22
Copper	7211	MET-23
Lead	742 1	MET-13
Selenium	7740	MET-14
Silver	7761	MET-1 8
Thallium	7841	MET-16
FIMS - Hg	7470A, 7471A	MET-57

Metals Analysis - Instrument Operating Procedures

	EPA-600	SW-846	
ICP - AES	200.7	601 OB	MET-27
ICP - MS	200.8	6020	MET-58
GFAA - 51002	- -	7000	MET-l 0
FIMS - Hg	-----	7470A, 7471A	MET-57

Volatiles Analysis

	EPA-600	SW-846	
Volatile GC/MS Analysis Low Level GC/MS Analysis	- -	524.2	VOA-6
Volatile GC/MS Analysis	- - -	82608	VOA-5
Waters and Methanol Extracted Solids	- - -	5035	
Volatile GC/MS Analysis Solid matrices	- - -----	82608 5030B	VOA-9

SemiVolatile Extractions

AQUEOUS

APPENDIX E
LABORATORY CAPABILITIES

	METHOD REFERENCE		EN CHEM SOP
	EPA-600	SW-846	
Base/ Neutral/ Acids - Water	USEPA 40CFR 625	3510C	SVO-1
Base/ Neutral/ Acids - Continuous Liauid-Liauid Extraction		3520C	svo-3
Base/ Neutral/ Acids - TCLP	USEPA 57CFR 227	351oc	svo-2
Organochlorine Pesticides/PCBs		3510C	SVO-6
Organochlorine Pesticides - TCLP		351oc	svo-7
Organochlorine Pesticides/PCBs - CLP	-----	USEPA SOW'	SVO-8
Chlorinated Herbicides		81508	svo-13
Chlorinated Herbicides - TCLP		81508	svo-14
Total Petroleum Hydrocarbons by Infrared Spectroscopy	418.1		SVO-23
Polynuclear Aromatic Hydrocarbons by HPLC		351 OB, 36308	SVO-40

*USEPA Statement of Work for Organics Analysis, Document # OLM03.1
CFR : USEPA Code of Federal Regulations

COMPOSITIONAL

	EPA-600	SW-846	
Base/ Neutral/ Acids - Soil		3550B	SVO-4
Base/ Neutral/ Acids - CLP		USEPA SOW"	SVO-5
Organochlorine Pesticides/PCBs		3550B	svo-10
Organochlorine Pesticides/PCBs - CLP		USEPA SOW*	SVO-11
Base/ Neutral/ Acids - Medium Level		3550A	svo-19
Base/ Neutral/ Acids - Medium Level - CLP	-----	USEPA SOW'	SVO-20
Toxaphene and PCBs - Medium Level		3550A	svo-2 1
Total Petroleum Hydrocarbons by Infrared Spectroscopy	418.1	- - -	SVO-24
Polynuclear Aromatic Hydrocarbons by H-PLC		35508	svo-39
Chlorinated Herbicides	----	8151	SVO-42
PCB Wipe Samples	- - -	3550A	svo-45
Organochlorine Pesticides/PCBs - Biological	- -	3550B	SVO-60
Polynuclear Aromatic Hydrocarbons - Biological samples	- - - -	354oc 3630C	SVO-6 1

^USEPA Statement of Work for Organics Analysis, Document # OLM01.8
*USEPA Statement of Work for Organics Analysis, Document # OLM03.1

SemiVolatile Analysis

	EPA-600	SW-846	
Chlorinated Herbicides		8000A, 8150A	SVO-32
Toxaohene		8000B 8081A	SVO-33

APPENDIX E
LABORATORY CAPABILITIES

	METHOD REFERENCE		EN CHEM SOP
Organochlorine Pesticides/PCBs - CLP	----	USEPA SOW"	SVO-36
Base/ Neutral/ Acids - GC/MS	USEPA 57CFR 625	80008. 8270C	svo-37
Base/ Neutral/ Acids - GC/MS - CLP	----	USEPA SOW^	SVO-38
Polynuclear Aromatic Hydrocarbons by HPLC		8000B, 8310	svo-41
Polynuclear Aromatic Hydrocarbons by HPLC	USEPA 40CFR 610	----	SVO-49
Methanol		8015A	svo-47
Nitroglycerin in Soil		8330	svo-50
Organochlorine Pesticides - GC		8000B, 8081A	SVO-51
Polychlorinated Biphenyls - GC		8000B, 8082	svo-52
Lipids Determination - Tissues, Plants, Fats	(Env.Tox.& Chem.')	(SM5520)	svo-59
Congener Specific PCB Determination - Capillary Column GC - Tissues	USEPA 40CFR 136	8081A, 8082, 3540C 3640A, 3630C,3660B	SVO-62
Congener Specific PCB Determination - Capillary Column GC - Soils	USEPA 40CFR 136	8081A, 8082, 3540C 3640A. 3630C,3660B	SVO-64

^USEPA Statement of Work for Organics Analysis, Document # OLM01.8
CFR : USEPA Code of Federal Regulations
*Evaluation of Selected Lipid Methods for Normalizing Pollutant Bioaccumulation, Environmental Toxicology and Chemistry, Vol 10, 1992
SM: Standard Methods for the Evaluation of Water and Wastewater, 1992

Other Capabilities

CLEANUP

	EPA-600	SW-846	
Florisil Cleanup	- -	USEPA SOW"	SVO-25
Sulfur Cleanup	- -	3660A	SVO-27
Sulfuric Acid Cleanup		3665A	SVO-28
Florisil Cleanup - PCBs	----.	36208	svo-57
Silica Gel Cleanup and Separation - Organochlorine Pesticides & PCBs	- -	36306	SVO-58

^USEPA Statement of Work for Organics Analysis, Document # OLM01.8

APPENDIX F

LABORATORY INSTRUMENT INVENTORY

LABORATORY INSTRUMENTATION

INSTRUMENT INVENTORY

Section Instrument/Peripherals	Date of Purchase
METALS	
1. ICP TJA 61 E TRACE ANALYZER	3100
2. ICP TJA 61 E TRACE ANALYZER	10198
3. GFAA (51002-2) (Graphite Furnace)	9/92
4. Perkin Elmer FIMS (Flow Inj. Mercury System)	8/98
5. Leeman Labs PS 200 Mercury Analyzer	1 *f94*
6. ICP-MS, Hewlet-Packard 4500 Series	*1199*
7. ICP-MS, Hewlet-Packard 4500 Series	3/00
SEMIVOLATILES	
1. Semivolatilesf Base/Neutral/Acid Extractables a. GCfMS HP 5973 w/Chemstation b. GCfMS HP 5972 w/Chemstation	 6/99 6/96
2. PESTICIDE a. GC/ECD(Dual) HP5890 (5 instruments) HP5890 Series 2 (6 instruments) b. GC/ECD(Dual) HP 6890	 1990-l 999 (various dates) *8198*
3. PESTICIDE/PCB CONGENERS a. *GCfMS* HP 5973 MSD *wf* APEX lg. volume injector.	 *1199*
3. TPH a. GC/FID HP5880 b. Mattson FT-IR	 1984 1989
4. EXTRACTIONS a. GPC (2, ABC 1002B), (2, 0.1. Autoprep 1000) b. Sonifier Cell Disruptors (4) c. Automated. Solvent Extractor d. ASP FP-41 Muffle Furnace	 1991- 2000 (various dates) 3/88 12/98 1988
5. HPLC a. PE 250 Binary LC pump – LC 240 Fluorescence Detector – 235 C Diode Array Detector – LC 101 oven b. PE 410 LC Pump – PE ISS 200 Advanced Sample processor – PE LC-95 UV/VIS Detector – McPherson FL-750 Spectro Fluorescence Detector – Pickering PCX 5100 Post Column Reactor Module – Eppendorf CH-30 Column Heater	 11/95 1986

INSTRUMENT INVENTORY (CONT.)

Section Instrument/Peripherals	Date of Purchase
VOLATILES	
1. Mass Spec 5971 HP (Mass Selective Detector, MSD) a. Tekmar Purge and Trap 2000/Dynatech PTA-30	6193 6/93
2. Mass Spec 5970 HP (MSD) a. Tekmar Purge and Trap 3000/Dynatech - Archon	5/91 5/96
3. Mass Spec 5973 HP (MSD) a. Tekmark Purge and Trap 3000/Dynatech - Archon	1/99 5/96
4. Mass Spec 5972 HP (MSD) a. Tekmar Purge and Trap 3000/Dynatech - Archon	6/96 6196
5. Mass Spec 5972 HP (MSD) a. Tekmar Purge and Trap 3000/Dynatech - Archon	6/96 6/96
6. Mass Spec 5973 HP (MSD) a. Tekmar Purge and Trap 3000/Dynatech - Archon	3/00 1/99
7. Chromatography Processing Systems a. HP Chemserver Data System	5/95 1/95
WET CHEMISTRY	
1. Total Organic Carbon Analyzer (TOC) Dohrmann DC-190 (2 instruments)	4/97, 7/99
2. Lachat Autoanalyzer	6/95
3. Spectrophotometer (UV-VIS SPEC)	3/86
4. Dionex DX-120 Ion Chromatograph	8/98

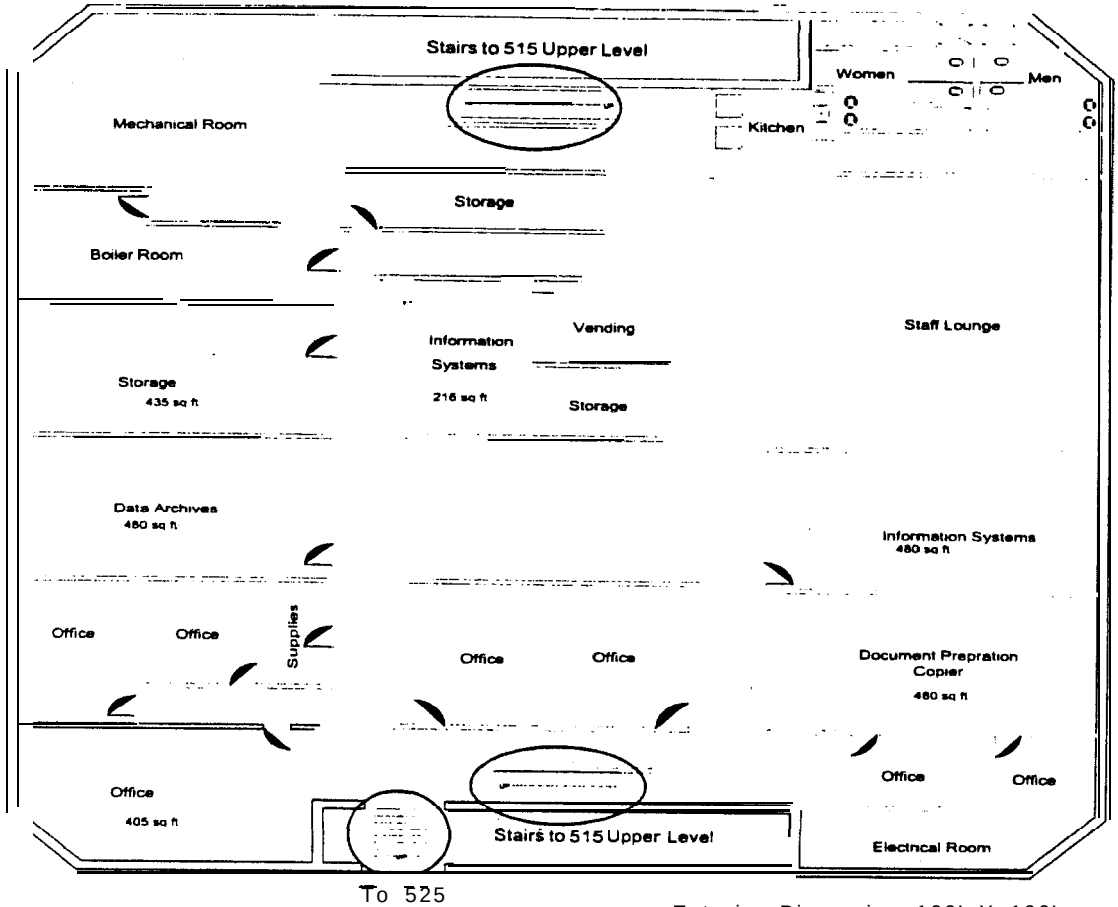
INSTRUMENT INVENTORY (CONT.)	
Section Instrument/Peripherals	**Date of Purchase**
SAMPLE PREP/OTHER	
1. TCLP Leach Tumblers non-volatile (3)	1990, 1996, 1998
2. TCLP ZHE Tumblers (2)	1990
3. pH Meters (2)	1986, 1999
4. Hot Plates (4)	various
5. Analytical Balances Mettler AE1 60 Mettler AJ 100 Mettler H35AR	various
6. Top Loading Balances (4)	various
7. Drying Ovens (6)	various
8. Constant Temperature Water Baths (2)	2000
9. Fluoride Probe, Orion 96-09	1993
10. Questron 3000 (microwave digestion)	6196
11. Mididistillation System (4 units)	6196 - 7/99
12. Tecator 2040 Digestor (block)	6197
13. Conductivity Meter, Accumet model 30	1/97
14. Hot Block Metals Digestion System (2 units)	1999
( ) = Number available in Laboratory.	

APPENDIX G

LABORATORY FLOORPLAN

En Chem, Inc.
515 Science Drive
Lower Level

Entrances/Exits

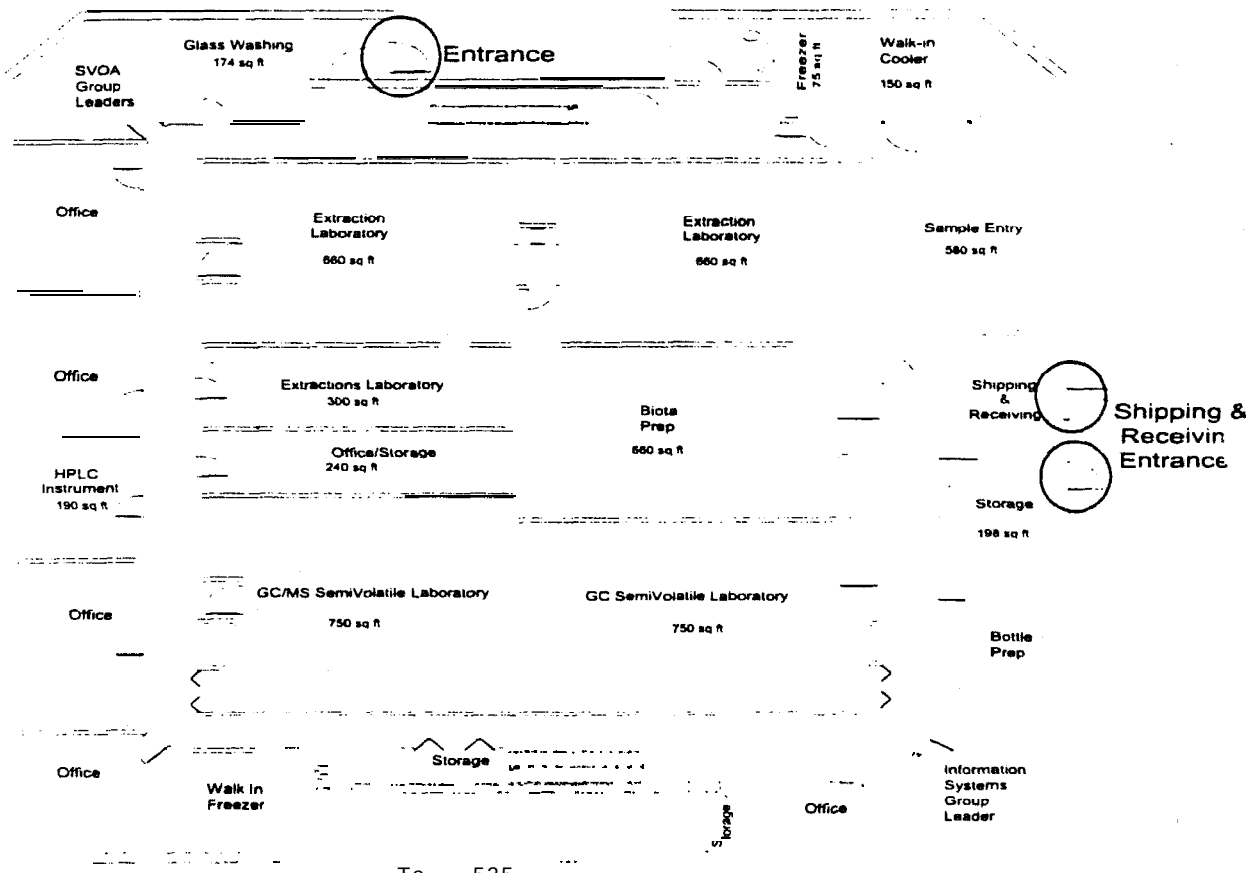


To 525
Lower Level

Exterior Dimension 100' X 100'
Scale 1/16" = 1'

En Chem, inc.
515 Science Drive
Upper Level

Entrances/Exits



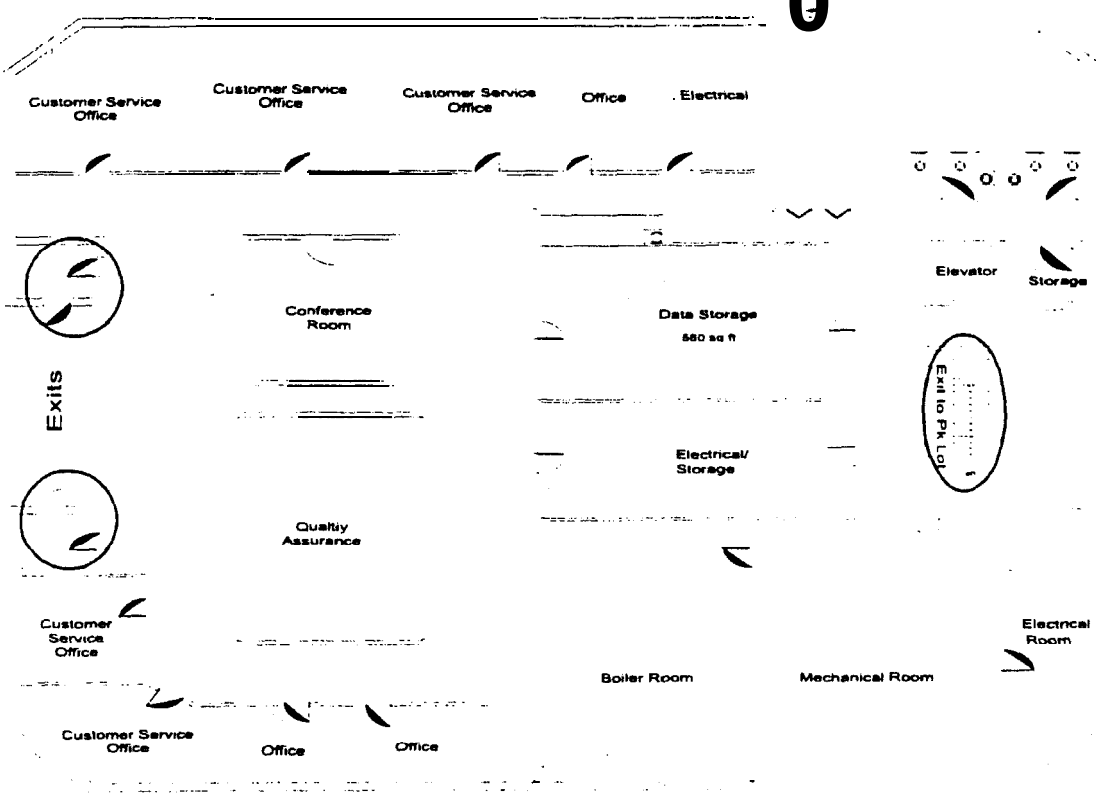
To 525
Upper Level

Exterior Dimension 100' X 100
Scale 1/16" = 1'

En Chem, Inc.
525 Science Drive
Lower Level

Entrance/Exits

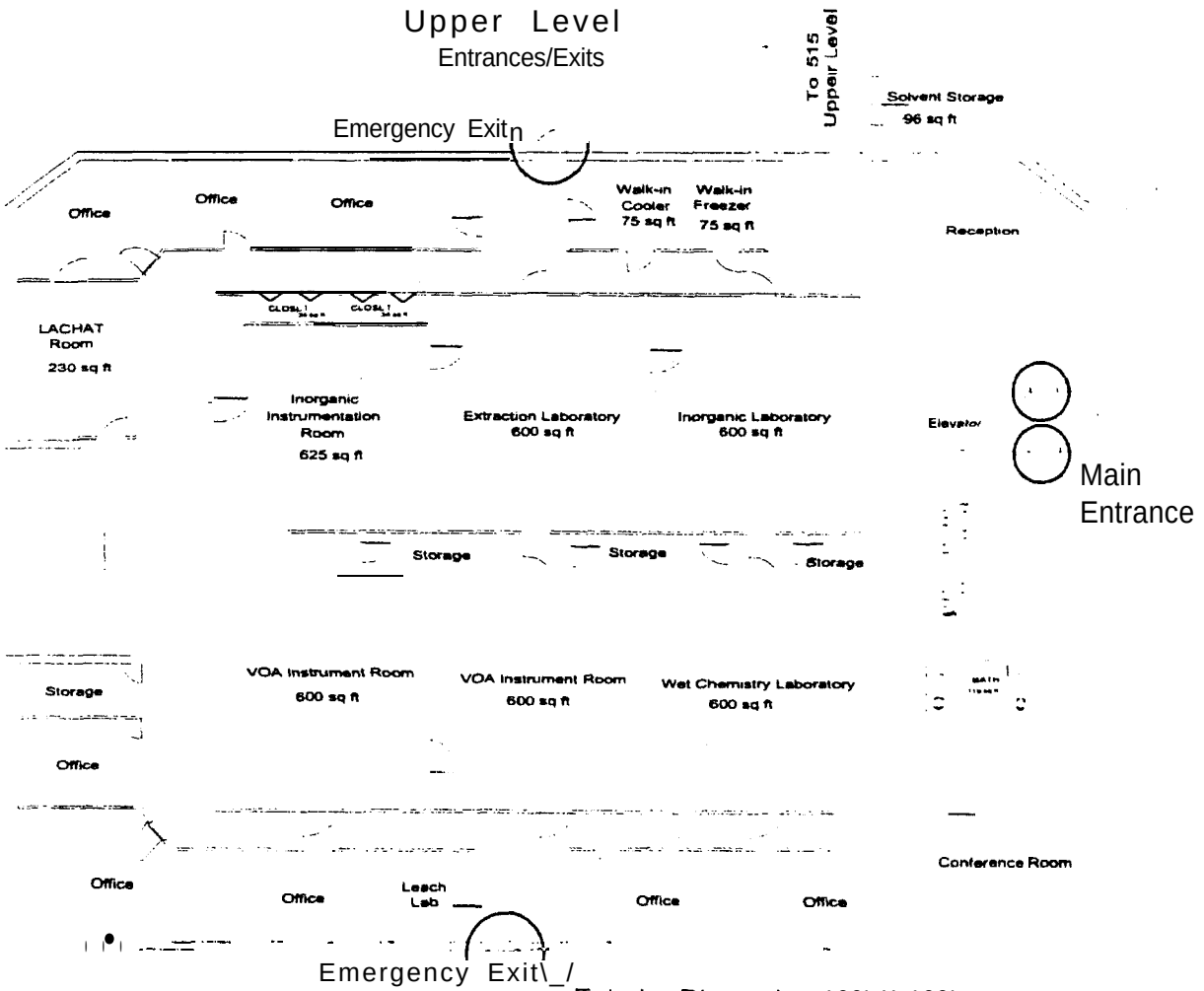
To 515
Lower Level



Exterior Dimension 100' X 100

Scale **1/16"** = 1'

En Chem, Inc.
525 Science Drive
Upper Level
Entrances/Exits



Exterior Dimension 100' X 100'
Scale **1/16"** = 1'

APPENDIX H

LABORATORY CERTIFICATIONS/APPROVALS

The State Of Wisconsin

DEPARTMENT OF NATURAL RESOURCES

Hereby grants

Certification

under the provisions of ch. NR 149, Wisconsin Administrative Code to:

En Chem, Inc. (Science Drive)
525 Science Drive
Madison, WI 53711

113172950
Laboratory ID Number

Issued: August 16, 2000

Expires: Auaust31, 2001

for the following test categories:

- Nitrogen
  - Ammonia as N
  - Nltrlte as N
  - Nitrate as N
  - Total Kjeldahl Nitrogen
- Phosphorus
  - Orthophosphate
  - Total Phosphorus
- Physical
  - Oil and Grease (Freon)
  - Total Dissolved Solids
  - Total Solids
  - Total Suspended Solids
  - Total Volatile Solids
- [illegible]
  - Alkalinity/Acidity
  - Bromide
  - Color
  - Hardness
  - Silica
  - Sulfite
- General II
  - Chloride
  - Cyanide
  - Chemical Oxygen Demand
  - Fluoride
  - Total Phenolic Compounds
- General II
  - Sulfide
  - Sulfate
- General III
  - EP Toxicity
  - Ignitability
  - Total Releasable Cyanide
  - Reactivity
  - Total Releasable Sulfide
  - SPLP
  - TCLP
  - Total Organic Carbon
  - Total Organic Halides
- Metals I
  - Silver
  - Aluminum
  - Arsenic
  - Boron
  - Barium
  - Beryllium
  - Calcium
  - Cadmium
  - Cobalt
  - Chromium (Total)
  - Copper
  - Iron
  - Chromium (Hexavalent)
  - Mercury
- Metals I
  - Potassium
  - Magnesium
  - Manganese
  - Molybdenum
  - Sodium
  - Nickel
  - Lead
  - Antimony
  - Selenium
  - Tin
  - Thallium
  - Vanadlum
  - Zinc
- Organics; Purgeable
  - Purgeable Aromatics
  - Purgeable Halocarbons
  - Volatile Organlcs (VOCs)
- Semivolatiles by GC/MS
  - Base/Neutral/Acid Extract
- Liquid Chromatography
  - PAHs by LC
  - Substituted Ureas
- Pesticides by GC
  - Acid Herbiddes
- Petroleum Hydrocarbons
  - Petroleum VOCs
- Organics; Organochlorine
  - PCBs
  - Organochlorine Pestiddes
- Safe Drinklng Water
  - Arsenic
  - Barium
  - Cadmium
  - Chmmlum
  - Copper
  - Mercury
  - Nitrate + Nitrite
  - Sodium
  - Nitrite
  - Nitrate
  - PAHs by HPLC
  - Lead
  - Selenium
  - Total Trihaiomethanes
  - Volatile Organics

Chief, Analytical and Statistical Services

George E. Meyer
Secretary

Certification or registration by the State of Wisconsin is not an endorsement or guarantee of the validity of data generated by this laboratory. This certificate is valid unless revoked or suspended and supersedes all previous certificates.

Rev. 3-96

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01, 2001
issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Lew of New York State

MR. DAVID E. TURRIFF
EN CHEM /NC
525 SCIENCE DRIVE
MADISON WI 53711 USA

NY Lab Id No: 1 7436
EPA Lab Code: WI00051

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Drinking Water Metals I	
Arsenic, *Total*	**EPA 200.8**
Barium, Total	**EPA 200.8**
Cadmium, Total	**EPA 200.8**
Chromium, Total	**EPA 200.8**
Copper, Total	**EPA 200.8**
Iron, Total	**EPA 200.7**
Lead, Total	**EPA 200.8**
Manganese, Total	**EPA 200.8**
Mercury, Total	EPA 200.8
Selenium, Total	**EPA 200.8**
Silver, Total	**EPA 200.8**
Sodium, Total	**EPA 200.7**
Zinc, Total	**EPA 200.8**

Drinking Water Metals II	
Antimony, Total	EPA 200.8
Beryllium, Total	EPA 200.8
Nickel, Total	**EPA 200.8**
Thallium, Total	EPA 200.8

Drinking Water Non-Metals	
Chloride	**EPA 300.0**
Cyanide	**EPA 335.4**
Fluoride, Total	**EPA 300.0**
Nitrate (as N)	EPA 300.0

Volatile Aromatics	
1,2,3-Trichlorobenzene	**EPA 524.2**
1,2,4-Trichlorobenzene	**EPA 524.2**
1,2,4-Trimethylbenzene	EPA524.2
1,2-Dichlorobenzene	**EPA 524.2**
1,3,5-Trimethylbenzene	**EPA 524.2**
1,3-Dichlorobenzene	EPA 524.2
1,4-Dichlorobenzene	**EPA 524.2**
2-Chlorotoluene	**EPA 524.2**
4-Chlorotoluene	**EPA 524.2**
Benzene	**EPA 524.2**
Bromobenzene	**EPA 524.2**
Chlombemene	**EPA 524.2**
Ethyl benzene	**EPA 524.2**
Hexachlombutadiene	**EPA 524.2**
Isopmpylbenzene	**EPA 524.2**
m-Xylene	**EPA 524.2**
n-Butylbenzene	**EPA 524.2**
o-Xylene	**EPA 524.2**
p-Isopropyltoluene (P-Cymene	EPA 524.2
p-Xylene	**EPA 524.2**
sec-Butylbenzene	**EPA 524.2**
Styrene	**EPA 524.2**
tert-Butylbenzene	**EPA 524.2**
Toluene	**EPA 524.2**

Serial No.: 10615

Property of the New York State Department of Health. Valid only at the address shown. Must be conspicuously posted. Valid certificates have a raised seal and may be verified by calling (518)485-5570.

DOH-3317 (3/97)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01, 2001
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DAVID E. TURRIFF
EN CHEM INC
525 SCIENCE DRIVE
MADISON WI 53771 USA

NY Lab Id No: 11436
EPA Lab Code: WI00051

is hereby APPROVED as an Environmental **Laboratory** *in conformance with the National Environmental Laboratory* **Accreditation** *Conference Standards for the category ENVIRONMEN-TAL ANALYSES POTABLE WATER*
All approved analytes are listed below:

Volatile Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 524.2
1,1,1-Trichloroethane	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 524.2
1,1,2-Trichloroethane	EPA 524.2
1,1-Dichloroethane	EPA 524.2
1,1-Dichloroethene	EPA 524.2
1,1-Dichloropropene	EPA 524.2
1,2,3-Trichloropropane	EPA 524.2
1,2-Dichloroethane	EPA 524.2
1 ,2-Dichloropropane	EPA 524.2
1,3-Dichloropropane	EPA 524.2
2,2-Dichloropropane	EPA 524.2
Bromochlommethane	EPA 524.2
Bromomethane	EPA 524.2
Carbon tetrachloride	EPA 524.2
Chloroethane	EPA 524.2
Chloromethane	EPA 524.2
cis-1,2-Dichloroethene	EPA 524.2
cis-1,3-Dichloropropene	EPA 524.2
Dibromomethane	EPA 524.2
Dichlorodifluoromethane	EPA 524.2
Methylene chloride	EPA 524.2
Tetrachloroethene	EPA 524.2
trans-1,2-Dichloroethene	EPA 524.2

Volatile Halocarbons	
trans-1,3-Dichloropropene	EPA 524.2
Trichloroethene	EPA 524.2
Trichlomfluoromethane	EPA 524.2
Vinyl chloride	EPA 524.2

Serial No.: 10615

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ooH-3317 (3/97)

NEW **YORK STATE DEPARTMENT OF HEALTH**
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01, 2001
Issued February 07, 2001

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Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DAVID E. TURRIFF
EN CHEM INC
525 SCIENCE DRIVE
MADISON Wi 53711 USA

NY Lab id No: 11436
EPA Lab Code: WI00051

is hereby APPROVED as an Environmental Laboratory for the category
ENVIRONMENTAL ANALYSES POTABLE WATER
Ail approved subcategories and/or anaiytes are listed below:

Drinking Water Non-Metals

Nitrite (as N)	EPA 300.0

Volatile Aromatics

p-lsopropyltoluene (P-Cymene'	Method Not Specified

Serial No.: 10616

DOH-3317 (3/97)

NEW **YORK STATE DEPARTMENT OF HEALTH**
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01, 2001
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DAVID E. TURRIFF
EN CHEM INC
525 SCIENCE DRIVE
MADISON WI 53711 USA

NY Lab Id No: 11436
EPA Lab Code: WI00051

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the **category**
ENVIRONMENTAL ANALYSES NON POTABLE WATER
Ail approved analytes are listed below:

Acrolein and Acrylonitrile	
Acrolein	EPA 624
Acrylonitrile	EPA 624
Benzidines	
3,3 dichlorobenzidine	EPA 625
Benzidine	EPA 625
Chlorinated Hydrocarbon Pesticides	
4,4-DDE	EPA 606
4,4-DDT	EPA 608
Aldrin	EPA 608
beta-BHC	EPA 608
Chlordane Total	EPA 608
delta-BHC	EPA 608
Endosulfan II	EPA 608
Endrin	EPA 608
Heptachlor	EPA 608
Methoxychlor	SM18 6630C
Chlorinated Hydrocarbons	
1,2,4-Trichlorobenzene	EPA 625
2-Chloronaphthalene	EPA 625
Hexachlorabenzene	EPA 625
Hexachlorobutadiene	EPA 625
Hexachlorocyclopentadiene	EPA 625
Hexachloroethane	EPA 625

Chlorophenoxy Acid Pesticides	
2.4,5-T	SM18 66408
2,4,5-TP (Silvex)	SM18 6640B
2.4-D	SM18 6640B
Haloethers	
4-Bromophenylphenyl ether	EPA 625
4-Chlorophenylphenyl ether	EPA 625
Bis (2-chloroisopropyl) ether	EPA 625
Bis(2-chloroethoxy)methane	EPA 625
Bis(2-chloroethyl)ether	EPA 625
Mineral	
Acidity	EPA 1979,305.1
Alkalinity	EPA 310.2
Chloride	EPA 300.0
	EPA 325.1
Hardness, Total	EPA 200.7
Sulfate (as SO4)	EPA 300.0
	EPA 375.1 & 375.2
Nitroaromatics and Isophorone	
2,4-Dinitrotoluene	EPA 625
2,6-Dinitrotoluene	EPA 625
Isophorone	EPA 625
Nitrobenzene	EPA 625

Serial No.: 10617

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DOH-3317 (3/97)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr. P.H. Commissioner

Expires 12:01 AM April 01, 2001
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance *with* and pursuant to section 502 *Public Health* Law *of New York State*

MR. DAVID E. TURRIFF
EN CHEM INC
525 *SCIENCE DRIVE*
MADISON WI 5371 1 USA

NY Lab Id No: 11436
EPA Lab Code: WI00051

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Nitrosoamines	
N-Nitrosodimethylamine	EPA 625
N-Nitrosodi-n-propylamine	EPA 625
N-Nitrosodiphenylamine	EPA 625
Nutrient	
Ammonia (as N)	EPA 350.1 EPA 350.3
Kjeldahl Nitrogen, Total	EPA 351.1 EPA 300.0
Nitrate (as N)	EPA 353.2 & 353.3
Phosphorus, Total	EPA 365.1
Phthalate Esters	
Benzyl butyl phthalate	EPA 625
Bis(2-ethylhexyl) phthalate	EPA 625
Diethyl phthalate	EPA 625
Dimethyl phthalate	EPA 625
Di-n-butyl phthalate	EPA 625
Di-n-octyl phthalate	EPA 625
Polychlorinated Biphenyls	
PCB-1232	EPA 606
PCB-1260	EPA 606
Polynuclear Aromatics	
Acenaphthene	EPA 601

Polynuclear Aromatics	
Acenaphthene	EPA 625
Anthracene	EPA 601
	EPA 625
Benzo(a)anthracene	EPA(610)
	EPA 625
Benzo(a)pyrene	EPA (610)
	EPA 625
Benzo(b)fluoranthene	EPA 601
	EPA 625
Fluoranthene	EPA (610)
	EPA 625
Indeno(1,2,3-cd)pyrene	EPA (610)
	EPA 625
Pyrene	EPA (610)
	EPA 625
Priority Pollutant Phenols	
2,4,5-Trichlorophenol	SW-646 8270C
2,4,6-Trichlorophenol	EPA 625
2,4-Dichlorophenol	EPA 625
2,4-Dimethylphenol	EPA 625
2,4-Dinitrophenol	EPA 625
2-Chlorophenol	EPA 625
2-Methyl-4,6-dinitrophenol	EPA 625

Serial No.: 10617

DOH-3317 (3/97)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01, 2001
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York state

MR. DAVID E. TURRIFF
EN CHEM INC
525 SCIENCE DRIVE
MADISON WI 53711 USA

NY Lab Id No: 17436
EPA Lab Code: WI00051

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Priority Pollutant Phenols	
2-Nitrophenol	EPA 625
4-Chloro-3-methylphenol	EPA 625
4-Nitrophenol	EPA 625
Pentachlorophenol	EPA 625
Phenol	EPA 625
Purgeable **Aromatics**	
1,3-Dichlorobenzene	EPA 624
	EPA 625
Benzene	EPA 624
Chlorobenzene	EPA 624
Ethyl benzene	EPA 624
Toluene	EPA 624
Total Xylenes	EPA 624
Purgeable **Halocarbons**	
1,1,1-Trichloroethane	EPA 624
1,1,2,2-Tetrachloroethane	EPA 624
1,1-Dichloroethene	EPA 624
1,2-Dichloroethane	EPA 624
Bromomethane	EPA 624
Carbontetrachloride	EPA 624
Chloroethane	EPA 624
Chloroform	EPA 624
Dibromochloromethane	EPA 624

Purgeable Halocarbons	
Methylenechloride	EPA 624
Trichloroethene	EPA 624
Trichlomfluommethane	**EPA 624**
Vinyl chloride	**EPA 624**
Resldoe	
Solids, Total	EPA 160.3
Solids, Total Dissolved	EPA 160.1
Solids, Total Suspended	EPA 160.2
Wastewater Metals I	
Barium. Total	EPA 200.7
	EPA 200.6
	MICROWAVE P/DIGEST
Cadmium, Total	EPA 200.7
	EPA 200.6
	EPA 200.9
	MICROWAVE P/DIGEST
Calcium, Total	**EPA 200.7**
Chmmium, Total	EPA 200.7
	EPA 200.6
	EPA 200.9
Copper, Total	EPA 200.7
	EPA 200.6
	EPA 200.9

Serial No.: 10617

DOH-3317 (3/97)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H. *Commissioner*

Expires 12:01 AM April 01, 2001
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued **in** *accordance* ***with*** *end* ***pursuant to section 502*** **Public** ***Health*** **Law of New York Slate**

MR. DAVID E. TURRIFF
EN CHEM INC
525 SCIENCE DRIVE
MADISON WI 53711 USA

NY Lab *Id No: 11436*
EPA Lab **Code:** *WI00051*

is hereby APPROVED **as** *an Environmental Laboratory in* ***conformance*** *with the National Environmental Laboratory Accreditation Conference* ***Standards*** *for the category* ***ENVIRONMENTAL*** *ANALYSES NON POTABLE WATER*
All approved analytes are listed below:

Wastewater Metals I	
Copper, Total	**MICROWAVE P/DIGEST**
Iron, Total	**EPA 200.7**
	MICROWAVE P/DIGEST
Lead, Total	EPA 200.7
	EPA200.8
	EPA 200.9
	MICROWAVE P/DIGEST
Magnesium, Total	EPA 200.7
Manganese, Total	EPA 200.7
	EPA 200.8
	MICROWAVE P/DIGEST
Nickel, Total	**EPA 200.7**
	EPA 200.8
	MICROWAVE P/DIGEST
Potassium, Total	EPA 200.7
Silver, Total	**EPA 200.7**
	EPA 200.8
	EPA 200.9
Sodium, Total	EPA 200.7
Wastewater Metals II	
Aluminum, Total	EPA 200.7
	EPA 200.8
	MICROWAVE **P/DIGEST**

Wastswater Metals II	
Antimony, Total	**EPA 200.7**
	EPA 200.8
	EPA 200.9
	MICROWAVE P/DIGEST
Arsenic, Total	**EPA 200.7**
	EPA 200.8
	EPA 200.9
	MICROWAVE P/DIGEST
Beryllium, Total	**EPA 200.7**
	EPA 200.8
Mercury, Total	**EPA 200.8**
	EPA 245.1
Selenium, Total	**EPA200.7**
	EPA 200.6
	EPA 270.2
	MICROWAVE P/DIGEST
Vanadium, Total	**EPA 200.7**
	EPA 200.8
Zinc. Total	EPA 200.7
	EPA 200.8
	MICROWAVE P/DIGEST
Wastewater Metals III	
Cobalt, Total	**EPA 200.7**

Serial No.: 10617

roperty of the New York State Departmen! of Health. Valid **only** at the address shown. Must be conspicuously posted. Valid certificates have a **raised** seal and may be verified by calling (518)485-5570.

DOH-3317 (3/97)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr. P.H. Commissioner

Expires 12:01 AM April 01, 2031
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DAVID E. TURRIFF
EN CHEM INC
525 SCIENCE DRIVE
MADISON WI 53711 USA

NY Lab Id No: 71436
EPA Lab Code: WI00057

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Wastewater Metals III

Cobalt, Total	EPA 200.8
Molybdenum, Total	EPA 200.7
Thallium, Total	EPA 200.7
	EPA 200.8
	EPA 200.9
Tin, Total	EPA 200.7
Titanium, Total	EPA 200.7

Wastewater Miscellaneous

Bromide	EPA 300.1
Color	EPA 110.2
Cyanide, Total	LACHAT 10-204-00-I-A
Oil & Grease Total Recoverabl	EPA 1664-A
Phenols	EPA 420.1
Specific Conductance	EPA 120.1
Sulfide (as S)	EPA 376.1

Serial No.: 10617

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DOH-3317 (3/97)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novel/o, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01, 2001
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DAVID E. TURRIFF
EN CHEM INC
525 SCIENCE DRIVE
MADISON WI 53711 USA

NY Lab Id No: 11436
EPA Lab Code: WI00051

is hereby APPROVED as an Environmental Laboratory for the category
ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved subcategories and/or analytes are listed below:

Chlorinated Hydrocarbon Pesticides	
4,4-DDD	EPA 606
alpha-BHC	EPA 608
Dieldrin	EPA 608
Endosulfan I	EPA 606
Endosulfan sulfate	EPA 606
Endrin aldehyde	EPA 606
Heptachlor epoxide	EPA 606
Lindane	EPA 606
Toxaphene	EPA 606
Chlorophenoxy Acid Pesticides	
Dicamba	Method Not Specified
Nutrient	
Nitrite (as N)	EPA 300.0
	EPA 354.1
Polychlorinated Biphenyls	
PCB-1016	EPA 606
PCB-1221	EPA 606
PCB-1242	EPA 606
PCB-1248	EPA 606
PCB-1254	EPA 608
Polynuclear Aromatics	
Acenaphthylene	EPA 601
Polynuclear Aromatics	
Acenaphthylene	EPA 625
Benzo(ghi)perylene	EPA 625
Benzo(k)fluoranthene	EPA 625
Chrysene	EPA (610)
	EPA 625
Dibenzo(a,h)anthracene	EPA (610)
	EPA 625
Fluorene	EPA (610)
	EPA 625
Naphthalene	EPA (610)
	EPA 625
Phenanthrene	EPA 601
	EPA 625
Purgeable Aromatics	
1,2-Dichlorobenzene	EPA 624
	EPA 625
1,4-Dichlorobenzene	EPA 624
	EPA 625
Purgeable Halocarbons	
1,1,2-Trichloroethane	EPA 624
1,1-Dichloroethane	EPA 624
1,2-Dichloroethene (total)	EPA 624
1,2-Dichloropropane	EPA 624

Serial No.: 10618

DOH-3317 (3/97)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. **Novello,** *M.D., M.P.H., Dr.P.H. Commissioner*

Expires 12:01 AM April 01, 2001
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance *with and pursuant to section* **502** *Public Health* **Law** *of New York State*

MR. DAVID E. TURRIFF
EN CHEM INC
525 SCIENCE DRIVE
MADISON WI 53717 USA

NY Lab Id No: 11436
EPA Lab Code: WI00051

is hereby APPROVED as an Environmental Laboratory for the category
ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved subcategories and/or analytes are listed below:

Purgeable **Halocarbons**	
2-Chloroethylvinylether	**Method Not Specified**
Bromodichloromethane	EPA 624
Bromoform	EPA 624
Chlommethane	EPA 624
cis-1,3-Dichloropropene	EPA 624
Dichlorodifluoromethane	Method Not Specified
Tetrachloroethene	EPA 624
trans-1,3-Dichloropropene	EPA 624
TCLP Additional Compounds	
Cresd	SW-@46 8270C
Methylethyl ketone (2-butanone	SW-846 8260B
Pyridine	SW-846 8270C
Wastewater Metals II	
Chromium VI	SM 18 3500-CrD

Serial No.: 10618

Property of the New York State Department of Health. Valid only at the address shown. Must be conspicuously posted. Valid certificates have a raised **seal** and may be verified by calling (518)485-5570.

DOH-3317 (3/97)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01,200;
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DAVID E. TURRIFF
EN CHEM INC
525 SCIENCE DRIVE
MADISON WI 53711 USA

NY Lab *id No: 71436*
EPA Lab Code: *WI00051*

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Acrolein and Acrylonitrile

Acrolein	SW-646 8260B
Acrylonitrile	SW-646 8260B

Characteristic Testing

TCLP	FED REG 1311

Chlorinated Hydrocarbon Pesticides

4,4-DDD	SW-646 8081A
alpha-BHC	SW-646 8081A
Chlordane Total	SW-646 8081A
Dieldrin	SW-846 8081A
Endosulfan I	SW-646 6081A
Endosulfan sulfate	SW-646 8081A
Endrin aldehyde	SW-646 8081A
Heptachlor epoxide	SW-846 8081A
Lindane	SW-646 8081A

Chlorinated Hydrocarbons

1,2,4-Trichlorobenzene	SW-646 627OC
2-Chloronaphthalene	SW-646 8270C
Hexachlorobenzene	SW-646 627OC
Hexachlombutadiene	SW-846 8270C
Hexachlorocyclopentadiene	SW-646 8270C
Hexachlomethane	SW-646 8270C

Chlorophenoxy Acid Pesticides

2,4,5-T	SW646 8151-A
2,4,5-TP (Silvex)	SW646 8151-A
2,4-D	SW646 8151-A
Dicamba	SW646 61514

Haloethers

Bis (2-chloroisopropyl) ether	SW-646 8270C
Bis(2-chloroethoxy)methane	SW-646 6270C

Metals I

Barium, Total	SW846 3005A
	SW646 3010A
	SW646 30508
	SW-W 3051
Cadmium, Total	SW-646 3020-A
	SW646 3005A
	SW646 3010A
	SW-646 3015
	SW646 30508
	SW-646 3051
Chromium, Total	SW-646 3020-A
	SW646 3005A
	SW646 301 OA
	SW-646 3015
	SW646 30508

Serial No.: 10619

Property of the New York State Department of Health. Valid only at the address shown. Must be conspicuously posted. Valid certificates have a raised seal and may be verified by calling (518)485-5570.

DOH-3317 (3/97)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Anfonia C. Novello, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01,200:
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with *and* ***pursuant*** **to section 502** *Public Health Law of New York State*

MR. DAVID E. TURRIFF
EN CHEM INC
525 SCIENCE DRIVE
MADISON WI 53711 USA

NY Lab *Id No: 11436*
EPA Lab Code: WI00051

is hereby APPROVED as an ***Environmental*** *Laboratory in conformance with* ***the*** *National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE All approved analyfes are listed below:*

Metals I

Chromium, Total	SW-8463051
Lead, Total	SW-646 3020-A
	SW646 3005A
	SW646 3010A
	SW846 30508
	SW-646 3051
	SW-646 601 0B
	SW-646 6020
Nickel, Total	SW-646 3020-A
	SW646 3005A
	SW646 3010A
	SW-646 3015
	SW646 30508
	SW-646 3051
	SW-646 6010B
	SW-846 6020
Silver, Total	SW646 3005A
	SW6463015
	SW646 30508
	SW-646 60108
	SW-646 6020
	SW-646 7472

Metals II

Antimony, Total	SW646 3005A
	SW-646 3015
	SW646 3050B
	SW-646 601 OB
	SW-646 6020
	SW646 7041
Arsenic, Total	SW646 3005A
	SW646 3010A
	SW646 30508
	SW-846 3051
	SW-646 601OB
	SW-646 6020
	SW646 7060A
Selenium, Total	SW646 3005A
	SW-646 3051
	SW-846 6010B
	SW-646 6020
	SW-646 7740

Nitroaromatics and Isophorone

2,4-Dinitrotoluene	SW-646 6270C
2,6-Dinitrotoluene	SW-046 827OC
Nitrobenzene	SW-846 8270C

Serial No.:10619

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DOH-3317 (3/97)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novel/o, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01, 2001
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DAVID E. TURRIFF
EN CHEM INC
525 SCIENCE DRIVE
MADISON WI 53711 USA

NY Lab Id No: 1 7436
EPA **Lab Code:** *WI00051*

is hereby APPROVED es an Environmental Laboratory in **conformance** *with. the* **National** *Environmental Laboratorv Accreditation Conference Standards for the* **category** *ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE*
All approved analytes are listed below:

Phthalate Esters	
Benzyl butyl phthalate	SW-846 8270C
Bis(2-ethylhexyl) phthalate	SW-846 8270C
Diethyl phthalate	SW-846 827OC
Dimethyl phthalate	SW-S46 8270C
Di-n-butyl phthalate	SW-846 8270C
Di-n-octyl phthalate	SW-846 8270C
,lychlorinated Biphenyls	
PCB-1016	SW-846 8082
PCB-1221	SW-846 8082
PCB-1232	SW-846 8082
PCB-1242	SW-046 8082
PCB-1248	SW-046 8082
PCB-1254	SW-846 8082
PCB-1260	SW-846 8082
Polynuclear Aromatic Hydrocarbons	
Acenaphthylene	SW-846 8270C
	SW-046 8310
Benzo(b)fluoranthene	SW-046 8270C
	SW-846 8310
Benzo(ghi)perylene	SW-846 8270C
	SW-846 8310
Chrysene	SW-846 827OC
	SW-846 8310

Polynuclear Aromatic Hydrocarbons	
Dibenzo(a,h)anthracene	SW-846 8270C
	SW-846 8310
Fluorene	SW-846 8270C
	SW-846 8310
Naphthalena	SW-846 8270C
	SW-846 8310
Phenanthrene	SW-646 8270C
	SW-846 8310
Priority Pollutant Phenols	
2,4,6-Trichlorophenol	SW-846 827OC
2,4-Dichlorophenol	SW-846 827OC
2,4-Dimethylphenol	SW-846 8270C
2,4-Dinitrophenol	SW-846 827OC
2-Chlorophenol	SW-846 8270C
2-Methyl-4,6-dinitrophenol	SW-646 8270C
2-Nitrophenol	SW-846 827OC
4-Chloro-3-methylphenol	SW-846 827OC
4-Nitrophenol	SW-646 827OC
Pentachlorophenol	SW-846 8270C
Phenol	SW-046 8270C

Serial No.: 10619

DOH-3317 (3/97)

NEW **YORK STATE DEPARTMENT OF HEALTH**
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01, 2001
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DAVID E. TURRIFF
EN CHEM /NC
525 SCIENCE DRIVE
MADISON WI 53711 USA

NY Lab Id No: 11436
EPA Lab Code: WI00051

is hereby APPROVED as an Environmental Laboratory for the category
ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved subcategories and/or analytes are listed below:

Chlorinated Hydrocarbon Pesticides

4,4-DDE	SW-846 8081A
4,4-DDT	SW-646 8081A
Aldrin	Method Not Specified
beta-BHC	SW-646 808lA
delta-BHC	SW-846 8081Á
Endosulfan II	SW-846 8081A
Endrin	SW-846 8081A
Heptachlor	SW-646 8081A
Methoxychlor	SW646 8081A
Toxaphene	SW-646 8081A

Metals II

Chromium VI	SW-646 7196A
Merwry, Total	SW646 7470A
	SW646 7471A

Nitroaromatics and Isophorone

Isophorone	Method Not Specified

Polynuclear Aromatic Hydrocarbons

Acenaphthene	SW-846 8270C
	SW-646 8310
Anthracene	SW-646 8270C
	SW-646 8310
Benzo(a)anthracene	SW-646 8270C
	SW-646 8310

Polynuclear Aromatic Hydrocarbons

Benzo(a)pyrene	SW-346' 827OC
	SW-646 8310
Fluoranthene	SW-846 8270C
	SW-646 8310
Indeno(1,2,3-cd)pyrene	SW-646 8270C
	SW646 8310
Pyrene	SW646 8270C
	SW-646 8310

Purgeable Aromatics

1,2-Dichlorobenzene	SW-646 8260B
1,3-Dichlorobenzene	SW-646 8260B
1,4-Dichlorobenzene	SW-646 8260B
Benzene	SW-646 8260B
Chlorobenzene	SW-646 8260B
Ethyl benzene	SW-646 8260B
Toluene	SW-646 8260B
Total Xylenes	SW-646 8260B

Purgeable Halocarbons

1,1,1-Trichloroethane	SW-646 82608
1,1,2,2-Tetrachloroethane	SW646 8260B
1,1,2-Trichloroethane	SW-646 8260B
1,1-Dichloroethane	SW-646 8260B
1,1-Dichloroethene	SW-646 8260B

Serial No.: 10620

NEW YORK STATE DEPARTMENT OF HEALTH
. WADSWORTH **CENTER**
Antonia C. Novello, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01, 2001
Issued February 07, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DAVID E. TURRIFF
EN CHEM INC
525 SCIENCE DRIVE
MADISON WI 53711 USA

NY Lab Id No: 11436
EPA Lab Code: WI00051

is hereby APPROVED as en Environmental Laboratory for the category
ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved subcategories and/or analytes are listed below:

Purgeable Halocarbons

1,2-Dichloroethane	SW-646 8260B
1,2-Dichloropropane	SW-646 8260B
2-Chloroethylvinyl ether	SW-646 82608
Bromodichloromethane	SW-846 82608
Bmmoform	SW-646 8260B
Bromomethane	SW-646 82608
arbon tetrachloride	SW-646 82608
Chloroethane	SW-646 82608
Chloroform	SW-646 82608
Chloromethane	SW-646 8260B
Dibromochloromethane	SW-846 82608
Dichlorodifluoromethane	SW-846 8260B
Methylene chloride	SW-846 82608
Tetrachloroethene	SW-646 82608
Trichloroethene	SW-646 8260B
Trichlorofluoromethane	SW-846 8260B
Vinyl chloride	SW-646 8260B

Serial No.: 10620

State of Arkansas

Department of Environmental Quality
Laboratory Certification Program

Be it known that **En Chem, Inc.**
Madison, Wisconsin

has earned certification by this Department for the period of

June 30, 2000 . to June 15, 2001

The following parameters are certified:

Hardness	Copper	Silver
Aluminum	Iron	Sodium
Antimony	Lead	Thallium
Arsenic	Magnesium	Tin
Barium	Manganese	Titanium
Beryllium	Mercury	Vanadium
Cadmium	Molybdenum	Zinc
Calcium	Nickel	Pesticides and PCBs
Chromium	Potassium	Semi-volatiles
Cobalt	Selenium	Volatile Organics

June 30, 2000
Date

Jeffrey M. Luehr
Quality Assurance Officer

NORTH DAKOTA STATE DEPARTMENT OF HEALTH
RECOGNITION OF CERTIFICATION OR ACCREDITATION

This is to certify that the North Dakota State Department of Health recognizes the certification or accreditation of

En Chem, Inc. – Madison, Wisconsin

by the State of North Carolina Department of the Environment and Natural Resources

for: all wastewater/groundwater parameters for which they retain certification from the State of North Carolina Department of the Environment and Natural Resources Division of Water Quality Laboratory Certification Program

Certificate Number R-159 Date of issue Nov. 15, 1999 Expiration Date December 31, 2000

This certificate remains the property of the North Dakota State Department of Health and may be recalled for cause, at any time, by the department.

Director, Division of Chemistry

Certification Officer

D H E C

PROMOTE PROTECT PROSPER

South Carolina Department of Health and Environmental Control

Environmental Laboratory Certification Program

In accordance with the provisions of Regulation 61 - 81, entitled "State Environmental Laboratory Certification Regulation,"

EN CHEM INC - MADISON
525 SCIENCE DR
MADISON, WISCONSIN 53711

is hereby certified to perform analyses as documented on the attached parameter list(s). This certification does not guarantee validity of data generuted, but indicates the laboratory's adherence to prescribed methodology, quality control, records keeping, and reporting procedures. This certificate is the property of S.C. DHEC und must be surrendered upon demand. This certificate is non-trunsferuble and is valid only for the parumeters and methodology listed on the attached parameter list(s).

Laboratory Director: DAVID TURRIFF PHD
Certifying Authority: WI
Date of Issue: September 05, 2000
Date of Expiration: August 31, 2001
Certificate Number: 83001001

/Director
Office of Environmental Laboratory Certification

CR-000509 8/99

SOUTH CAROLINA DEPARTMENT OF HEALTH AND ENVIRONMENTAL CONTROL
ENVIRONMENTAL LABORATORY CERTIFICATION PROGRAM

EN CHEM INC -MADISON (Laboratory ID 83001)
Laboratory Director: DAVID TURRIFF PHD
Certifying Authority: WI
Certificate Number: 83001001

Date of Issue: September 05, 2000
Expiration Date: August 31, 2001

CLEAN WATER ACT

HERBICIDES

CHLORINATED PHENOXY ACID HERBICIDES	SM 6640B

INORGANIC - MINERAL

ACIDITY	EPA305.1
ALKALINITY	EPA 310.1
CHLORIDE	EPA325.3
FLUORIDE	EPA340.2
SULFATE	EPA 375.4

INORGANIC - MISCELLANEOUS

CYANIDE	EPA 335.3
OIL&GREASE	EPA 413.1
SULFIDE	EPA376.1

INORGANIC - NUTRIENT

AMMONIA-NITROGEN	EPA 350.2
KJELDAHL-NITROGEN	EPA351.3
NITRATE-NITRITE (N02&N03)	EPA353.2
NITRATE-NITROGEN	NO3-NO2 MINUS NO2
NITRITE-NITROGEN	EPA 354.1
PHOSPHORUS	EPA 365.2

INORGANIC - TRACE METAL

ALUMINUM	EPA 200.7
ANTIMONY	EPA204.2
ARSENIC	EPA200.7
BARIUM	EPA200.7
BERYLLIUM	EPA200.7
BORON	EPA200.7
CADMIUM	EPA200.7
CADMIUM	EPA213.2
CALCIUM	EPA200.7
CHROMIUM	EPA200.7
CHROMIUM	EPA218.2
CHROMIUM, HEXAVALENT	EPA 218.4
COBALT	EPA200.7
COPPER	EPA 200.7
IRON	EPA200.7
LEAD	EPA200.7
LEAD	EPA239.2
MAGNESIUM	EPA200.7
MANGANESE	EPA200.7

SOUTH *CAROLINA* DEPARTMENT OF HEALTH AND ENVIRONMENTAL CONTROL
ENVIRONMENTAL LABORATORY CERTIFICATION PROGRAM

EN CHEM INC - MADISON (Laboratory ID 83001)
Laboratory Director: DAVID TURRIFF PHD
Certifying Authority: WI
Certificate Number: 8300.2001

Date of Issue: September OS, 2000
Expiration Date: August 31, 2001

CLEAN WATER ACT

INORGANIC - TRACE METAL

MERCURY	EPA 245.1
MOLYBDENUM	EPA 200.7
NICKEL	EPA 200.7
POTASSIUM	EPA200.7
SELENIUM	EF'A200.7
SILICA, DISSOLVED	EPA200.7
SILVER	EPA 272.2
SODIUM	EPA200.7
THALLIUM	EPA279.2
VANADIUM	EPA200.7
ZINC	EPA200.7

PCBS AND PESTICIDES

ORGANOCHLORINE PESTICIDES & PCBS - GC/ECD	EPA608

SEMI-VOLATILES

BASE/NEUTRALS AND ACIDS - GC/MS	EPA 625
POLYNUCLEAR AROM. HYDROC. (PAHS) - GC/FID OR	EPA610

VOLATILES (VOCS)

PURGEABLES - GC/MS	EPA624

SOLID & HAZARDOUS WASTES

INORGANIC- DEMAND

TOTAL ORGANIC CARBON (TOC)	EPA9060

INORGANIC - HAZARDOUS WASTE CHARACTERISTICS

IGNITABILITY (SETAFLASH)	EPA 1020A
TCLP - BOTTLE EXTRACTION	EPA 1311
TCLP-ZEROHEADSPACE	EPA1311

INORGANIC - MINERAL

CHLORIDE .	EPA9250
SULFATE	EPA 9036

INORGANIC - MISCELLANEOUS

CYANIDE	EPA 9012A

SOUTH CAROLINA DEPARTMENT OF HEALTH AND ENVIRONMENTAL CONTROL
ENVIRONMENTAL LABORATORY CERTIFICATION PROGRAM

EN CHEM INC - MADISON (Laboratory ID 83001)
Laboratory Director: DAVID TURRIFF PHD
Certifying Authority: WI
Certificate Number: 83001001

Date of Issue: September 05, 2000
Expiration Date: August 31, 2001

SOLD & HAZARDOUS WASTES

INORGANIC - TRACE METAL

ALUMINUM	EPA 6010B
ANTIMONY	EPA7041
ARSENIC	EPA 6010B
ARSENIC	EPA 7060A
BERYLLIUM	EPA 6010B
CADMIUM	EPA 6010B
CADMIUM	EPA 7131A
CALCIUM	EPA 6010B
CHROMIUM	EPA 6010B
CHROMIUM, HEXAVALENT	EPA 7196A
COBALT	EPA 6010B
COPPER	EPA 6010B
IRON	EPA 6010B
LEAD	EPA7421
MAGNESIUM	EPA 6010B
MANGANESE	EPA 6010B
MERCURY	EPA 7470A
M O L Y B D E N U M	EPA 6010B
NICKEL	EPA 6010B
POTASSIUM	EPA 6010B
SELENIUM	EPA7740
SILVER	EPA 6010B
SODIUM	EPA 6010B
THALLIUM	EPA7841
VANADIUM	EPA 6010B
ZINC	EPA 6010B

PCBS AND PESTICIDES

ORGANOCHLORINE PESTICIDES BY GC: CAP.COLUMN	EPA 8081A	EPA 3510C
ORGANOCHLORINE PESTICIDES BY GC: CAP.COLUMN	EPA 8081A	EPA 3550B
POLYCHLORINATED BIPHENYLS BY GC	EPA8082	EPA 3510C
POLYCHLORINATED BIPHENYLS BY GC	EPA8082	EPA 3550B

SEMI-VOLATILES

POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC	EPA8310	EPA 3510C
POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC	EPA8310	EPA 3550B
SEMIVOLATILE ORGANICS BY GC/MS: CAPILLARY CO	EPA 8270C	EPA 3510C
SEMIVOLATILE ORGANICS BY GC/MS: CAPILLARY CO	EPA 8270C	EPA 3550B

VOLATILES (VOCS)

VOLATILE ORGANICS BY GC/MS: CAPILLARY COLUMN	EPA 8260B	EPA 5030B
VOLATILE ORGANICS BY GC/MS: CAPILLARY COLUMN	EPA 8260B	EPA5035

SOUTH CAROLINA DEPARTMENT OF HEALTH AND ENVIRONMENTAL CONTROL
ENVIRONMENTAL LABORATORY CERTIFICATION PROGRAM

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Certificate Number: 83001001

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CLEANWATERACT

----------HERBICIDES----------

SM 66408

2.4.5-T
2,4,5-TP (SILVEX)
2.4-D

--------PCBS AND PESTICIDES--------

EPA 608

4,4'-DDD
4,4'-DDE
4,4'-DDT
ALDRIN
ALPHA-BHC
BETA-BHC
CHLORDANE
DELTA-BHC
DIELDRIN
ENDOSULFAN I
ENDOSULFAN II
ENDOSULFAN SULFATE
ENDRIN
ENDRIN ALDEHYDP
GAMMA-BHC (LINDANE)
HPPTACHLOR
HEPTACHMR EPOXIDE
METHOXYCHLOR
PCB-1016 (AROCLOR-1016)
PCB-1221 (AROCLOR-12211
PCB-1232 (AROCLOR-1232)
PCB-1242 (AROCLOR-1242)
PCE-1248 (AROCLOR-1248)
PCB-1254 (AROCLOR-1254)
PCS-1260 (AROCLOR-1260)
TOXAPHENE

----------SEMI-VOLATILES----------

EPA 610

ACENAPHTHENE
ACENAPHTHYLENE
ANTHRACENB
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE

EPA 610

CHRYSENE
DIBENZO(A,H)ANTHRACENE
PLUORANTHENE
FLUORENE
INDENO(1,2,3-CD)PYRENE
NAPHTHALENE
PHENANTHRENE
PYRENE

EPA 625

1,2,4-TRICHLOROBENZENE
1,2-DICHLOROBENZENE
1,3-DICHLOROBBNZBNB
1,4-DICHLOROBENZENE
2,4,6-TRICHLOROPHENOL
2,4-DICHLOROPHENOL
2,4-DIMETHYLPHENOL
2,4-DINITROPHENOL
2,4-DINITROTOLUENE
2,6-DINITROTOLUENE
2-CHLORONAPHTHALENE
2-CHLOROPHENOL
2-METHYL-4,6-DINITROPHENOL
2-NITROPHENOL
3,3-DICHLOROBENZIDINE
4-BROMOPHENYLPHENYL ETHER
4-CHLORO-3-METHYLPHENOL
4-CHLOROPHENYL PHENYL BTHER
4-NITROPHENOL
ACENAPHTHENE
ACENAPHTHYLENE
ANTHRACENE
BENZIDINE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
BENZYL BUTYL PHTHALATE
BIS(2-CHLOROETHOXY)METHANE
BIS(2-CHLOROETHYL)ETHER
BIS(2-CHLOROISOPROPYL)ETHER
BIS(2-ETHYLHEXYL)PHTHALATE
CHRYSENE
DI-N-BUTYL PHTHALATE
DI-N-OCTYL PHTHALATE
DIBENZO(A,H)ANTHRACENE

EPA 625

DIMETHYL PHTHALATE
FLUORANTHENE
FLUORENE
HEXACHLOROBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROCYCLOPENTADIENE
HEXACHLOROETHANE
INDENO(1,2,3-CD)PYRENE
ISOPHORONE
N-NITROSODI-N-PROPYLAMIN%
N-NITROSODIMETHYLAWNE
N-NITROSODIPHENYLAMINE
NAPHTHALENE
NITROBENZENE
P%NTACHLOROPHBNOL
PHENANTHRENE
PHENOL
PYR%NE

----------VOLATILES (VOCS)----------

EPA 624

1,1,1-TRICHLOROETHANE
1,1,2,2-TETRACHLOROETHANE
1,1,2-TRICHLOROETHANE
1,1-DICHLOROETHANE
1,1-DICHLOROETHENE
1,2-DICHLOROBENZENE
1,2-DICHLOROETHANE
1,2-DICHLOROPROPANE
1,3-DICHLOROBENZENE
1,4-DICHLOROBENZENE
BENZENE
BROMODICHLOROMETHANE
BROMOFORM
BROMOMETHANE
CMBON TETRACHLORIDE
CHLOROBENZENE
CHMROETHANE
CHLOROFORM
CHLOROMETHANE
CIS-1,3-DICHLOROPROPENE
DIBROMOCHLOROMETHANB
DICHLORODIFLUOROMETHANE
ETHYLBENZENE
METHYLENE CHLORIDE
NAPHTHALENE

EPA 624

TOLUENE
TRANS-1,2-DICHLOROETHENB
TRANS-1,3-DICHLOROPROPENE
TRICHMROETHBNE
TRICHLOROFLUOROMETHANE
VINYL CHLORIDE
XYLENE, TOTAL

SOUTH CAROLINA DEPARTMENT OF HEALTH AND ENVIRONMENTAL CONTROL
ENVIRONMENTAL LABORATORY CERTIFICATION PROGRAM

EN CHEM INC - MADISON (Laboratory ID 830011
Certifying Authority: WI
Certificate Number: 83001001

Date of Issue: September 05, 2000
Expiration Date: Auguet 31, 2001

SOLID & HAZARDOUS WASTES

--------PCBS AND PESTICIDES------

EPA 8081A
EPA 3510C

4,4'-DDD
4,4'-DDE
4,4'-DDT
ALDRIN
ALPHA-BHC
ALPHA-CHLORDANE
BETA-BHC
CHLORDANE
DELTA-BHC
DIELDRIN
ENDOSULFAN I
BNDOSULFAN SULFATE
ENDRIN
ENDRIN ALDEHYDE
ENDRIN KETONE
GAMMA-EHC (LINOANE)
GAMMA-CHLORDANE
HEPTACHLOR
HEPTACHLOR EPOXIDE
NETHOXYCHLOR
TOXAPHENE

EPA 8081A
EPA 35508

4,4'-DDD
4,4'-DDE
4,4'-DDT
ALORIN
ALPHA-CHLORDANE
DELTA-BHC
DIELDRIN
ENDOSULFAN I
ENDOSULFAN II
ENDOSULFAN SULFATE
ENDRIN
ENDRIN ALDEHYDE
ENDRIN KETONE
GAMMA-BHC (LINDANE)
GAMMA-CHLORDANB
HEPTACHLOR
HEPTACHLOR EPOXIDE
METHOXYCHLOR
TOXAPHENE

EPA 8082
EPA 3510C

PCB-1016 (AROCMR-1016)
PCB-1221 (AROCLOR-1221)
PCB-1232 (AROCLOR-1232)
PCB-1242 (AROCLOR-1242)
PCB-1248 (AROCLOR-1248)
PCB-1254 (AROCLOR-1254)
PCB-1260 (AROCLOR-1260)

EPA 8082
EPA 3550B

PCB-1016 (AROCLOR-1016)
PCB-1221 (AROCLOR-1221)
PCB-1232 (AROCLOR-1232)
PCR-1242 (AROCLOR-1242)
PCE-1248 (AROCLOR-1240)
PCE-1254 (AROCLOR-1254)
PCB-3260 (AROCLOR-1260)

----------SEMI-VOLATILES------

EPA 8270C
EPA 3510C

1,2,4-TRICHLOROBENZENE
1,2-DICHLOROBENZENE
1,2-DIPHENYLHYDRAZINE
1,3-DICHLOROBENZENE
1,4-DICHLOROBENZENE
2,4,5-TRICHLOROPHENOL
2,4,6-TRICHLOROPHENOL
2,4-DICHLOROPHENOL
2,4-DIMETHYLPHENOL
2,4-DINITROPHENOL
2,4-DINITROTOLUENE
2,6-DINITROTOLUENE
2-CHLORONAPHTHALENE
2-CHLOROPHENOL
2-METHYLNAPHTHALENE
2-METHYLPHENOL
2-NITROANILINE
2-NITROPHENOL
3,3-DICHLOROBENZIDINE
3-METHYLPHENOL
3-NITROANILINE
4,6-DINITRO-2-METHYLPHENOL
4-BROMOPHENYLPHENYL ETHER
4-CHLORO-3-METHYLPHENOL

EPA 8270C
EPA 3510C

4-CHLOROANILINE
4-CHLOROPHENYL PHENYL ETHER
4-METHYLPHENOL
4-NITROANILINE
4-NITROPHENOL
ACENAPHTHBNE
ACENAPHTHYLENE
ACETOPHENONE
ANILINE
ANTHRACENE
BENZO(A)ANTHRACENE
BENZO(A) PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
FJENZOIC ACID
RENZYL ALCOHOL
BIS(2-CHLOROETHOXY)METHANE
BIS(2-CHLOROETHYL)ETHER
BIS(2-CHLOROISOPROPYL)ETHER
BIS(2-ETHYLHEXYL)PHTHALATE
BUTYL BENZYL PHTHALATE
CHRYSENE
DI-N-BUTYL PHTHALATE
DI-N-OCTYL PHTHALATE
DIBENZ(A,J)ACRIDINE
DIBENZO(A,H)ANTHRACENE
DIBENZOFURAN
DIETHYL PHTHALATE
DIMETHYL PHTHALATE
FLUORANTHENE
FLUORENE
HEXACHLOROBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROCYCLOPENTADIENE
HEXACHLOROETHANE
INDENO(1,2,3-CD)PYRENE
ISOPHORONE
N-NITROSODI-N-PROPYLAMINE
N-NITROSODIMETHYLAMINE
N-NITROSODIPHENYLAMINE
NAPHTHALENE
NITROBENZENE
PENTACHLOROPHENOL
PHENANTHRENE
-4NOL
ENE
.8IDINE

EPA 8270C
EPA 3550B

1,2,4-TRICHLOROBENZENE
1,2-DICHLOROBENZENE
1,2-DIPHENYLHYDRAZINE
1,3-DICHLOROBENZENE
1,4-DICHLOROBENZENE
2,4,5-TRICHLOROPHENOL
2,4,6-TRICHLOROPHENOL
2,4-DICHLOROPHENOL
2,4-DIMETHYLPHENOL
2,4-DINITROPHENOL
2,4-DINITROTOLUENE
2,6-DINITROTOLUENE
2-CHLORONAPHTHALENE
2-CHLOROPHENOL
2-METHYLNAPHTHALENE
2-METHYLPHENOL
2-NITROANILINE
2-NITROPHENOL
3,3-DICHLOROBENZIDINE
3-METHYLPHENOL
3-NITROANILINE
4,6-DINITRO-2-METHYLPHENOL
I-BROMOPHENYLPHENYL ETHER
4-CHLORO-3-METHYLPHENOL
4-CHLOROANILINE
4-CHLOROPHENYL PHENYL ETHER
4-METHYLPHENOL
4-NITROANILINE
4-NITROPHENOL
ACENAPHTHENE
ACENAPHTHYLENE
ACETOPHENONE
ANILINE
ANTHRACRNE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
BENZOIC ACID
BRNZYL ALCOHOL
BIS(2-CHLOROETHOXY)METHANE
BIS(2-CHLOROETHYL)ETHER
BIS(2-CHLOROISOPROPYL)ETHER
BIS(2-ETHYLHEXYL)PHTHALATE
BUTYL BENZYL PHTHALATE
CHRYSENE
DI-N-BUTYL PHTHALA'

SOUTH CAROLINA DEPARTMENT OF HEALTH AND ENVIRONMENTAL CONTROL
ENVIRONMENTAL LABORATORY CERTIFICATION PROGRAM

EN CHEM INC - MADISON (Laboratory ID 83001)
Certifying Authority: WI
Certificate Number: 83001001

Date of Issue: September 05, 2000
Expiration Date: August 31, 2001

SOLID & HAZARDOUS WASTES

----------SEMI-VOLATILES----------

EPA 8270C
EPA 35508

DI-N-OCTYL PHTHALATE
DIBENZ(A,J)ACRIDINE
DIBENZO(A,H)ANTHRACENE
DIBENZOFURAN
DIETHYL PHTHALATE
DIMETHYL PHTHALATB
FLUORANTHENE
FLUORENE
HEXACHLOROBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROCYCLOPENIENE
HEXACHLOROETHANE
INDENO(1,2,3-CD)PYRENE
ISOPHORONE
N-NITROSODI-N-PROPYLAMINE
N-NITROSODIHETHYLAMINE
N-NITROSODIPHENYLAMINE
NAPHTHALENE
NITROBENZENE
PENTACHLOROPHENOL
PHENANTHRENE
PHENOL
PYRENE
PYRIDINE

EPA 8310
EPA 3510C

ACENAPHTHENE
ACENAPHTHYLENE
ANTHRACENE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
CHRYSENE
DIBENZO(A,H)ANTHRACENE
FLUORANTHENE
FLUORENE
INDENO(1,2,3-CD)PYRENE
NAPHTHALENE
PHENANTHRENE
PYRENE

EPA 8310
EPA 35508

ACENAPHTHENE
ACENAPHTHYLENE
ANTHRACENE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
CHRYSENE
DIBENZO(A,H)ANTHRACENE
FLUORANTHENE
FLUORENE
INDENO(1,2,3-CD)PYRENE
NAPHTHALENE
PHENANTHRENE
PYRENE

---------VOLATILES (VOCS) --e-v-----

EPA 82608
EPA 5030B

1,1,1,2-TETRACHLOROETHANE
1,1,1-TRICHLOROETHANE
1,1,2,2-TETRACHLOROETHANE
1,1,2-TRICHLOROETHANE
1,1-DICHLOROETHANE
1,1-DICHLOROETHENE
1,1-DICHLOROPROPENE
1,2,3-TRICHLOROBENZENE
1,2,3-TRICHLOROPROPANE
1,2,4-TRICHLOROBENZENE
1,2,4-TRIMETHYLBENZENE
1,2-DIBROMO-3-CHLOROPROPANE(DBCP)
1,2-DIBROMOETHANE (EDB)
1,2-DICHLOROBENZENE
1,2-DICHLOROETHANE
1,2-DICHLOROPROPANE
1,3,5-TRIMETHYLBENZENE
1,3-DICHLOROBENZENE
1,3-DICHLOROPROPANE
1,4-DICHLOROBENZENE
2,2-DICHLOROPROPANE
2-CHLOROETHYLVINYL ETHER
2-CHLOROTOLUENE
2-HEXANONE
4-CHLOROTOLUENE

EPA 8260B
EPA 50308

ACETONE
ACROLEIN
ACRYLONITRILE
ALLYL CHLORIDE
BENZENE
BROMOBENZENE
BROMOCHLOROMETHANE
BROMODICHLOROMETHANE
EROMOFORM
BROMOMETHANE
CARBON DISULFIDE
CARBON TETRACHLORIDE
CHLOROBBNZENE
CHLOROETHANE
CHLOROFORM
CHLOROMETHANE
CIS-1,2-DICHLOROETHENE
CIS-1,3-DICHLOROPROPENE
CIS-1,4-DICHLORO-2-BUTENE
DIBROMOCHLOROMETHANE
DIBROMOFLUOROMETHANE
DIBROMOMETHANE
DICHLORODIFLUOROMETHANE
DIETHYL ETHER
ETHYL METHACRYLATE
ETHYLBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROETHANE
IODOMETHANE
ISOPROPYLBENZENE
METHYL ETHYL KETONE (MEK)
METHYL METHACRYLATE
METHYL TERT BUTYL ETHER (MTBE)
METHYLENE CHLORIDE
N-BUTYLBENZENE
N-PROPYLBLNZBNR
NAPHTHALENE
P-ISOPROPYLTOLUENE
SEC-BUTYLBENZENE
STYRENE
TERT-BUTYLBENZENE
TETRACHLOROETHENE
TOLUENE
TRANS-1,2-DICHLOROETHENE
TRANS-1,3-DICHLOROPROPENE
TRANS-1,4-DICHLORO-2-BUTENE
TRICHLOROETHENE

EPA 8260B
EPA 5030B

VINYL ACETATE
VINYL CHLORIDE
XYLENE. TOTAL

EPA 8260B
EPA 5035

1,1,1,2-TETRACHLOROETHANE
1,1,1-TRICHLOROETHANE
1,1,2,2-TETRACHLOROETHANE
1,1,2-TRICHLOROETHANE
1,1-DICHLOROETHANE
1,1-DICHLOROETHENE
1,1-DICHLOROPROPENE
1,2,3-TRICHLOROBENZENE
1,2,3-TRICHLOROPROPANE
1,2,4-TRICHLOROBENZENE
1,2,4-TRIMETHYLBENZENE
1,2-DIBROMO-3-CHLOROPROPANE(DBCP)
1,2-DIBROMOETHANE (EDB)
1,2-DICHLOROBENZENE
1,2-DICHLOROETHANE
1,2-DICHLOROPROPANE
1,3,5-TRIMETHYLBENZENE
1,3-DICHLOROBENZENE
1,3-DICHLOROPROPANE
1,4-DICHLOROBENZENE
2,2-DICHLOROPROPANE
2-CHLOROETHYLVINYL ETHER
2-CHLOROTOLUENE
2-HEXANONE
4-CHLOROTOLUENE
4-METHYL-2-PENTANONE
ACETONE
ACROLEIN
ACRYLONITRILE
ALLYL CHLORIDE
BENZENE
BROMORENXENE
BROMOCHLOROMETHANE
BROMODICHLOROMETHANE
BROMOFORM
BROMOMETHANE
CARSON DISULFIDE
CARBON TETRACHLORIDE
CHLOROBENZENE
CHLOROETHANE

SOUTH CAROLINA DEPARTMENT OF HEALTH AND ENVIRONMENTAL CONTROL
ENVIRONMENTAL LABORATORY CERTIFICATION PROGRAM

EN CHEM INC - MADISON (Laboratory ID 83001)
Certifying Authority: WI
Certificate Number: 83001001

Date of Issue: September 05, 2000
Expiration Date: August 31, 2001

SOLID & I IAZARDOUS WASTES

----------VOLATILES (VOCS)----------

EPA 8260B
EPA 5035

CHLOROMBTHANE
CIS-1,2-DICHLOROETHENE
CIS-1,3-DICHLOROPROPENE
CIS-1,4-DICHLORO-2-BUTENE
DIBROMOCHLOROMETHANE
DIBROMOFLUOROMETHANE
DIBROMOMETHANE
DICHLORODIFLUOROMETHANE
DIETHYL ETHER
ETHYLBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROETHANE
IODOMETHANE
ISOPROPYLBENZENE
METHACRYMNITRILE
HETHYL KTHYL KBTONK (MEK)
METHYL TERT BUTYL ETHER (MTBE)
METHYLENE CHLORIDE
N-BUTYLBENZENE
N-PROPYLBENZENE
NAPHTHALENE
P-ISOPROPYLTOLUENB
PENTACHLOROETHANE
SEC-BUTYLBENZENE
STYRENE
TERT-BUTYLBENZENE
TETRACHLOROETHENE
TOLUENK
TRANS-l,Z-DICHLOROETHENE
TRANS-1,3-DICHLOROPROPBNE
TRANS-1,4-DICHLORO-2-BUTENE
.TRICHLOROKTHKNE
TRICHMROFLUOROMBTHANE
VINYL ACETATE
VINYL CHLORIDE
XYLENE, TOTAL

STATE OF NORTH CAROLINA DEPARTMENT OF THE
ENVIRONMENT AND NATURAL RESOURCES

DIVISION OF WATER QUALITY
LABORATORY CERTIFICATION PROGRAM

In *accordance with the provisions of N.C.G.S.* ***143-215.3*** *(a)* ***(1), 143-215.3 (a)(10)*** *and NCAC* ***2H.0800:***

EN CHEM, INC.

Is hereby certified to perform environmental analysis **as** *listed on Attachment I and* ***report*** *monitoring data to* ***DWQ*** *for compliance* ***with*** *NPDES* ***effluent,*** *surface water,* ***groundwater,*** *and pretreatment* ***regulations.***

By reference ***15A*** *NCAC 2H .0800 is made* **a** *part of this* ***certificate.***

This *certificate does not guarantee* **validity of** *data* ***generated,*** *but* **indicates** *the methodology, equipment, quality control procedures, records, and proficiency of the laboratory have been examined* **and** *found to be acceptable.*

This ***certificate*** *shall be valid until* **December 31. 2003**

Certificate No. 503

James W Meyer
James W. Meyer

Attachment I

North Carolina Wastewater/Groundwater Laboratory Certification

Certified Parameters Listing

Lab Name: EN CHEM, Inc.

Address: 525 Science Drive
Madison, WI 53711-

Certificate Number: 503
Effective Date: 01/01/2001
Expiration Date: 12/31/2003
Date of Last Amendment: 01/17/2001

The above named laboratory, having duly met the requirements of 15A NCAC 2H.0800, is hereby certified for the measurement of the parameters listed below.

CERTIFIED PARAMETERS

INORGANIC
COD
CHLORIDE
CHLORIDE
CYANIDE
FLUORIDE
FLUORIDE
HARDNESS, TOTAL
METALS I, REGULAR LEVEL
ALUMINUM
ARSENIC
BERYLLIUM
CADMIUM
CHROMIUM, TOTAL
COBALT
COPPER
IRON
LEAD
MANGANESE
NICKEL
SELENIUM
VANADIUM
ZINC
METALS II. REGULAR LEVEL
ANTIMONY
SILVER
THALLIUM
METALS I, LOW LEVEL
ARSENIC
CADMIUM
CHROMIUM, TOTAL
COPPER
LEAD
SELENIUM
METALS II, LOW LEVEL
ANTIMONY
SILVER
THALLIUM
BARIUM
MERCURY
TOTAL KJELDAHL NITROGEN
NO2 + NO3 NITROGEN
NITRATE
NITRATE NITROGEN
NITRITE
-RITE NITROGEN
AL PHOSPHORUS
OIL & GREASE - 413.1
OIL & GREASE - EPA 1664
OIL & GREASE - EPA 9071A
pH
INORGANIC PHENOLS
RESIDUE, TOTAL
RESIDUE, DISSOLVED 180 C
RESIDUE, SUSPENDED
SULFATE
SULFATE
TOTAL ORGANIC CARBON
TURBIDITY
TCLP
METALS
ORGANICS

ORGANIC
ORGANOCHLORINE PESTICIDES & PCBs
EPA 608
ORGANOCHLORINE PESTICIDES
EPA 8081A + 3500 SERIES
POLYCHLORINATED BIPHENYLS (PCB'S)
EPA 8082 + 3500 SERIES
PURGEABLE ORGANICS
EPA 624
EPA 5030 + 8260B
BASE NEUTRAL/ACID ORGANICS
EPA 625
EPA 8270C + 3500 SERIES
CHLORINATED ACID HERBICIDES
EPA 8151A + 3500 SERIES

This certification requires maintance of an acceptable quality assurance program, use of approved methodology, and satisfactory performance on evaluation samples. Laboratories are subject to civil penalties and/or decertification for infractions as set forth in 15A NCAC 2H.0807.

MINNESOTA
MDH
DEPARTMENT OF HEALTH

Public Health Laboratory

Minnesota Department of Health

Certificate

In accordance with Minnesota Law and Rules

En Chem, Inc. - Madison
525 Science Drive, Madison, Wisconsin 53711
Laboratory Number: 055-999-107

has been certified for the analytes listed below and in our letter dated October 6, 2000.

Clean Water Program
Inorganics
Metals
Volatile Organic Compounds
Synthetic Organic Compounds

Safe Drinking Water Program
Inorganics
Metals
Volatile Organic Compounds

Resource Conservation and Recovery Program
Metals
Toxicity Characteristic Leaching Procedure
Volatile Organic Compounds
Synthetic Organic Compounds

Certification Expiration Date: August 3 1.2002

Jan Malcolm, Commissioner of Health

MINNESOTA
MDH
DEPARTMENT OF HEALTH

Protecting, maintaining and improving the health of all Minnesotans

October 6, 2000

Gregory J. Graf
En Chem, Inc. - Madison
525 Science Drive
Madison, Wisconsin 537 11

RE: Laboratory Number 055-999-107

Dear Mr. Graf:

We have received your laboratory's Application for Certification, appropriate fees, performance evaluation results and Wisconsin Certificate of Approval. After reviewing all the information/documents received, we are issuing certification for the analytes as listed below in accordance with the reciprocity agreement between the states of Wisconsinand Minnesota.

Laboratory Number 055-999-107 Certification Expiration Date: 31-AUG-2002

Clean Water Program

Alkalinity
Chemical Oxygen Demand
Chloride
Cyanide
Nitrogen, Ammonia
Nitrogen, Total Kjeldahl
Nitrogen, Nitrate
Nitrogen, Nitrate + Nitrite
Nitrogen, Nitrite
Oil and Grease
Organic Carbon, Total
Phenol, Total Compounds
Phosphorus, Ortho
Phosphorus, Total
Solids, Dissolved
Solids, Nonfilterable (TSS)
Sulfate
Sulfide
Aluminum
Antimony
Arsenic
Barium
Beryllium
Cadmium
Calcium
Chromium, Hexavalent
Chromium
Cobalt
Copper
Iron
Lead
Magnesium
Manganese
Mercury
Nickel
Potassium
Selenium
Silver
Sodium
Thallium
Vanadium
Zinc
Dichlorodifluoromethane
Chloromethane
Vinyl chloride
Bromomethane
Chloroethane
Trichlorofluoromethanc
1,1-Dichloroethene
Methyl&e chloride
trans-1,2-Dichloroethene
1,1-Dichloroethane
2,2-Dichloropropane
cis-1,2-Dichloroethene
Chloroform
Bromochloromethane
1,1,1-Trichloroethane
Carbon tetrachloride
1,2-Dichloroethane
Trichloroethcne
1,2-Dichloropropane
Bromodichloromethane
Dibromomethane
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
1,1,2-Trichloroethane
1,3-Dichloropropane

Clean Water Program (continued)

Tetrachloroethtne
Chlorodibromomethane
1,2-Dibromoethane
Chlorobenzene
1,1,1,2-Tetrachloroethane
Bromoform
1,1,2,2-Tetrachloroethane
1,2,3-Trichloropropane
Bromobenzene
2-Chlorotoluene
4-Chlorotoluene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
1,2-Dibromo-3-Chloropropane
1,2,4-Trichlorobenzene
Hexachlorobutadiene
1,2,3-Trichlorobenzene
Benzene
Toluene
Ethyl benzene
m+p-Xylene
o-Xyiene
Styrene
Isopropylbenzene
n-Propylbenzene
1,3,5-Trimethylbenzene
tert-Butylbenzene
1,2,4-Trimethylbenzene
set-Butylbenzene
p-Isopropyltoluene
n-Butylbenzene
Naphthaiene
Acetone
Ethyl ether
2-Chloroethylvinylether
Methyl ethyl ketone
Butyl benzyl phthalate

Di-2(ethylhexyl) phthalate
Di-n-butyl phthalate
Di-n-octyl phthalate
Diethyl phthalate
Dimethyl phthalate
Aldrin
alpha-BHC
beta-BHC
delta-BHC
gamma-BHC (Lindane)
Chlordane
4,4'-DDD
4,4'-DDE
4,4'-DDT
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan sulfate
Endrin
Endrin Aldehyde
Heptachlor
Heptachlor epoxide
PCB-1016
PCB-1221
PCB-1232
PCB-1242
PCB-1248
PCB-1254
PCB-1260
Toxaphene
4-Chloro-3-methylphenol
2-Chlorophenol
2,4-Dichlorophenol
2,4-Dimethylphenol
2,4-Dinitrophenol
2-Methyl-4,6-dinitrophenol
2-Nitrophenol
Pentachlorophenol

Phenol
2,4,6-Trichlorophenol
4-Nitrophenol
Bis-(2-chloroethyl) ether
Bis-(2-chloroethoxy)methane
4-Bromophenylphenyl ether
4-Chlorophenylphenyl ether
2-Chloronaphthalene
Hexachlorobenzene
Hexachlorobutadiene
Hexachlorocyclopentdiene
Hexachloroethane
1,2,4-Trichlorobenzene
Acenaphthene
Acenaphthylene
Anthracene
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(g,h,i)perylene
Benzo(k)fluoranthene
Chrysene
Dibenz(a,h)anthracene
Fluoranthene
Fluorene
Indeno(1,2,3-cd)pyrene
Phenanthrene
Pyrene
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Isophorone
Nitrobenzene
Benzidine
3,3'-Dichlorobenzidine
N-Nitrosodimethylamine
N-Nitrosodiphenylamine
N-Nitrosodi-n-propyiamine

Safe Drinking Water Program

Fluoride
Nitrogen, Nitrate
Nitrogen, Nitrite
Arsenic
Barium
Cadmium
chromium
Copper
Lead
Mercury
Selenium
Chloromethane
Vinyl chloride
Bromomethane
Chloroethane
1,1-Dichloroethene

Safe Drinking Water Program (continued)

Methylene chloride
trans-1,2-Dichloroethhene
1,1-Dichloroethane
2,2-Dichloropropane
cis-1,2-Dichloroethene
Chloroform
1,1,1-Trichloroethane
1,1-Dichloropropene
Carbon tetrachloride
1,2-Dichloroethane
Trichloroethene
1,2-Dichloropropane
Bromodichloromethane
Dibmmomethane
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
1,1,2-Trichloroethane
1,3-Dichloropropane
Tetrachloroethene
Chlorodibromomethane
1,2-Dibromoethane
Chlorobenzene
1,1,1,2-Tetrachloroethane
Bromoform
1,1,2,2-Tetrachloroethane
1,2,3-Trichloropropane
Bmmobenzene
2-Chlorotoluene
4-Chlorotoluene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
1,2-Dibromo-3-Chloropropane
1,2,4-Trichlorobenzene
Benzene
Tolucne
Ethyl benzene
m+p-Xylene
o-Xylene
Styrene
Isopropylbenzene

Resource Conservation and Recovery Program

Arsenic
Barium
Cadmium
chromium
Copper
Lead
Mercury
Molybdenum
Nickel
Selenium
Silver
Zinc
Toxicity Characteristic Leaching Proc
Acetone
Carbon disulfide
Ethyl ether
Methyl ethyl ketone
Methyl isobutyl ketone
Butyl benzyl phthalate
Di-2(ethylhexyl) phthalate
Di-n-butyl phthalate
Dimethyl phthalate
Alachlor
beta-BHC
gamma-BHC (Lindane)
4,4'-DDT
Endrin
PCBs
Toxaphene
2-Methylphenol
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Dibenz(a,h)anthracene
Fluoranthene
Indeno(1,2,3-cd)pyrene
Pyrene
B enzidine
1,2-Diphenylhydrazine
Benzoic acid

Enclosed is your laboratory's certificate.

If your laboratory wishes to renew its certification, send an application, appropriate fees, changes in your QA/Procedure Manual and most recent performance evaluation sample results to Certification, Public Health Laboratory, Minnesota Department of Health (MDH), 30 days prior to the expiration date noted above.

Your laboratory must analyze a performance evaluation sample from an approved provider for each certified analyte by August 31, 2001. The laboratory must forward the results of these performance evaluation samples to the MDH within 30 days from the date your laboratory receives them. In addition, it is the laboratory's duty to notify the MDH within 30 days of changes in laboratory location or ownership, major analytical equipment, test methodology or supervisory staff, as detailed in MN Rule 4740.2030, subpart 10.

If you have questions, please call Laboratory Accreditation at (612) 676-5200 or you may speak directly with one of the certification staff below:

Suzanne Skorich	(612) 676-5676
Susan Wyatt	(612) 676-5674

Sincerely,

Suzanne Skorich, Certification Officer
Environmental Laboratory Section
Public Health Laboratory Division
Minnesota Department of Health
P.O. Box 9441
Minneapolis, MN 55440-9441

SS:cs
Enclosure

Soil Permit

Permit Number: S-4879

UNITED STATES DEPARTMENT OF AGRICULTURE

Animal and Plant Health Inspection Service

Plant Protection and Quaranfine

Issued To: EnChem, inc.
(Gregory J. Graf)
802 Deming Way
Madison, Wisconsin 53717

TELEPHONE: (608) 827-5501

Under the authority of the Federal Plant Pest Act of May 23, 1957, permission is hereby granted to the facility/individual named above subject to the following conditions:

1. Valid for shipments of soil not heat treated at the port of entry, only if a **compliance agreement (PPQ Form 519) has been completed and signed.**
2. To be shipped in sturdy, leakproof, containers.
3. To be released without treatment at the port of entry.
4. To be used only for laboratory analysis, and only in the facility of the permittee at EnChem, Inc.. located in Madison, Wisconsin.
5. No use of soil for growing purposes is authorized, including the isolation or culture of organisms imported in soil.
6. All unconsumed soil, containers, and effluent is to be autoclaved, incinerated, or heat treated by the permittee at the conclusion of the **project as approved and prescribed by Plant Protection and Quarantine.**
7. This permit authorizes shipments from all foreign sources, including Guam, Hawaii, Puerto Rico, and the U.S. Virgin Islands through any U.S. port of entry staffed by Plant Protection and Quarantine.

DECEMBER 31.2001
Expiration Date

Approving Official DEBORAH M. KNOTT

DEPARTMENT OF THE ARMY
CORPS OF ENGINEERS
HTRW CENTER OF EXPERTISE
12565 WEST CENTER ROAD
OMAHA, NEBRASKA 68144-3869

REPLY TO
ATTENTION OF:

March 17, 1999

Hazardous, Toxic and Radioactive Waste
Center of Expertise

EnChem, Inc.
525 Science Drive
Madison, WI 53711-1060

Gentlemen:

This correspondence addresses the ongoing validation status of EnChem, Inc., of Madison, WI, for the U.S. Army Corps of Engineers (USACE) for chemical analysis in support of the USACE Hazardous, Toxic and Radioactive Waste Program.

EnChem, Inc., of Madison, WI, is now validated for the parameters listed below:

METHOD	-PARAMETERS	MATRIX"'
300 series	Anions[4]	Water[3]
8270C	Semivolatile Organics	Water[3]
8270C	Semivolatile Organics	Solids[3]
SW-846	TAL Metals[2]	Water"'
SW-846	TAL Metals[2]	Solids""
8081A	Pesticides	Water"'
8081A	Pesticides	Solids
8082	Polychlorinated Biphenyls	Water"'
8082	Polychlorinated Biphenyls	Solids[3]
Mod 8015	TPH - GRO/DRO	Water"'
Mod 8015	TPH - GRO/DRO	Solids"'
8260A	Volatile Organics	Water[3]
8260A	Volatile Organics	Solids

Remarks:

(1) 'Solids' includes soils, sediments, and solid waste.

(2) TAL Metals: Aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc.

(3) The laboratory has successfully analyzed a performance evaluation sample for this method/matrix.

(4) Anions: Chloride, fluoride, sulfate, nitrate, nitrite and ortho-phosphate.

(5) Approval for these parameters is based on review of SOPs only.

Based on the recent successful analysis for ortho-phosphate in water, Cobalt in soil and PCBs in soil, your laboratory will continue to be validated for sample analysis by the methods listed above. The period of validation for all parameters has been previously established and expires on June 3, 2000.

The USACE reserves the right to conduct additional laboratory inspections or to suspend validation status for any or all of the listed parameters if deemed necessary. It should be noted that your laboratory may not subcontract USACE analytical work to any other laboratory location without the approval of this office. This laboratory validation does not guarantee the delivery of any analytical samples from a USACE Contracting Officer Representative.

Any questions or comments can be directed to Richard Kissinger at (404) 697-2569. General questions regarding laboratory validation may be directed to the Laboratory Validation Coordinator at (402) 697-2574.

Sincerely,

for Marcia C. Davies, Ph.D.
Director, USACE Hazardous,
Toxic and Radioactive Waste
Center of Expertise

APPENDIX I

LABORATORY FORMS

Title

Form I-1 Madison Bottle Request Form

Form I-2 Chain of Custody Record

Form I-3 Subcontract Chain of Custody Form

Form I-4 Cooler Receipt Log

Form I-5 Madison Master Logbook Form

Form I-6 Madison Laboratory Tracking Sheet

Form I-7 Nonconformance Form

Form I-8 Sample Receiving Nonconformance Form

Madison Office & Laboratory
525 Science Drive
Madison, WI 53711
608-232-3300 . Fax: 608-233-0502
1-888-5-ENCHEM

EN CHEM INC.

Corporate Office & Laboratory
1795 Industrial Drive
Green Bay, WI 54302
[illegible] . Fax: 920-469-8827
1-800-7-ENCHEM

EN CHEM FIELD BOTTLE REQUEST FORM

Shipping Charge:_____

Client:_____
Project #:_____
Project Name:_____
SampleDate:_____
SampleType:_____

Date Bottles Needed:_____
Bottle Requestor:_____
Date Requested:_____

Bottle Preparer:_____
Date Prepared:_____
Checked By:_____

1) Is this a Wisconsin regulated project? Y N
2) Is there a residual chlorine in the water samples? Y N

Sample Points:

Parameters:

# of Bottles	Size	Type	Preservation	Associated Parameters	Special Instructions

Deliver to: _____
_____
_____
_____

Method of shipment:
_____

Number of coolers: _____

(Please Print Legibly)

Company Name _____

Branch or Location _____

Project Contact. _____

Telephone: _____

Project Number: _____

Project Name: _____

Project State: _____

Sampled By (Print) _____

EN CHEM INC.

1241 Bellevue St., Suite 9
Green Bay, WI 54302
920-469-2436
FAX 920-469 8827

525 Science Drive
Madison, WI 53711
608-232-3300
FAX: 608-233-0502

CHAIN OF CUSTODY

Page _____ of _____

P.O. # _____ Quote # _____

Mail Report To: _____

Company: _____

Address: _____

Invoice To: _____

Company: _____

Address: _____

Mail Invoice To: _____

*Preservation Codes
A=None B=HCL C=H2SO4 D=HNO3 E=EnCore F=Methanol G=NaOH
H = Sodium Bisulfate Solution I = Other

Data Package Options
(please circle if requested)
Results Only
EnChem Level III (Subject to Surcharge)
EnChem Level IV (Subject to Surcharge)

Regulatory Program
UST
RCRA
SDWA
NPDES
CERCLA

Matrix Codes
W=Water
S=Soil
A=Air
C=Charcoal
B=Biota
Sl=Sludge

LABORATORY ID (Lab Use Only)	FIELD ID	COLLECTION DATE	COLLECTION TIME	MATRIX	FILTERED? (YES/NO)	PRESERVATION (CODE)*	ANALYSES REQUESTED	TOTAL # OF BOTTLES SENT	CLIENT COMMENTS	LAB COMMENTS (Lab Use Only)

Rush Turnaround Time Requested (TAT) - Prelim
(Rush TAT subject to approval/surcharge)
Date Needed: _____
Transmit Prelim Rush Results by (circle):
Phone Fax E-Mail
Phone #: _____
Fax #: _____
E-Mail Address: _____

Sample special

HOLD are subject to and release of liability

Relinquished By:	Date/Time:	Received By:	Date/Time
Relinquished By:	Date/Time:	Received By:	Date/Time
Relinquished By:	Date/Time:	Received By:	Date/Time
Relinquished By:	Date/Time	Received By:	Date/Time
Relinquished By:	Date/Time	Received By	Date/Time

En Chem Project No.

Sample Receipt Temp.

Sample Receipt pH (Wet/Metals)

Cooler Custody Seal

Present / Not Present

Intact / Not Intact

EN CHEM INC.
chemistry for the environment

☐ 1241 Bellevue St., Green Bay, WI 54302
920-M-2436: FAX: 92046943827
☐ 525 Science Dr., Madison, WI 53711
608-232-3300;FAX:608-233-5052
☐ 1423 N. 8th Street, Superior. WI 54880
715-392-5844;FAX:715-392-5843

Chain of Custody

☐ Subcontract ☐ En Chem Internal Split

From: En Chem - _____

To: _____

_____

_____

Project Due: _____

☐ Normal Turn ☐ Quick Turn

Project State: _____

En Chem Project No.: _____

Requested Analysis

Filtered? YIN
Preservative

Preservation Code

A= None B=HCL
C= H2SO4 D=HNO3
E = EnCore F=Methanol
G= NaOH O=Other

En Chem Lab No.	Client Field I.D.	Date Sampled	Sample Type	No. of Bottles							Sub Lab Sample No.

Relinquished By:	Date/Time:	Received By:	Date/Time:
Relinquished By:	Date/Time:	Received By:	Date/Time:
Relinquished By:	Date/Time:	Received By:	Date/Time:

If you have questions, please contact _____ at: ☐ Green Bay ☐ Madison ☐ **Superior**

Please FAX/send **final** report to _____ at the: ☐ Green Bay ☐ **Madison** ☐ **Superior** Lab.

Final report to **be** generated by En **Chem:** ☐ Green Bay ☐ Madison ☐ **Superior**

COOLER RECEIPT LOG

Batch No._____

Project Name or ID_____ No. of Coolers:_____Temps:_____

A. Receipt Phase: Date cooler **was** opened:_____ By:_____

1: Were samples received **on** ice? (Must be ≤ 6 C)	YES	NO[2]	
2. Was there a Temp. Blank?.	YES	NO	
3: Were custody seals present and intact? (Record on COC).	YES	NO	
4: Are COC documents present?.	YES	NO'	
5: is this Project a Quick **Turn** Project?.	YES.	NO	
6: **Is** there any sub-work?.	YES	NO	
7: Are there any short hoidtime tests?.	YES	NO	
8: Are any samples nearing expiration of hold-time? (Within 2 days).	YES'	NO	Contacted **by/Who**_____
9: Do any samples need to be Filtered or Preserved in the lab?.	YES'	NO	Contacted **by/Who**:_____

B. Log-in Phase: Date samples were logged-in:_____ **By:**_____

1: Were **all** sample containers listed on the COC received and intact?.	YES	**NO[2]**	**NA**
2: Sign the COC as received by En Chem. Completed..	YES	**NO**	
3: Do sample labels match the COC?	YES	**NO[2]**	
4: Check sample **pH** of preserved samples. (not **VOCs)** Completed..	YES	**NO**	**NA**
5: Are sample volumes adequate for tests requested?	YES	**NO[2]**	
6: Are VOC samples free of bubbles **>6mm**	YES	**NO[2]**	NA
7: Enter samples into master logbook. Completed..	YES	NO	
8: Place laboratory sample number on all containers. Completed..	YES	NO	
9: Complete LTS sheet. Completed	YES	NO	
IO: Complete nonconformance record if applicable. Completed..	YES	NO	NA
11: Initiate Subcontracting procedure, SOP **1-REC-4,** if applicable. Completed	YES	NO	NA

Short Hold-time tests:

48 Hours or less	7 days	Footnotes
Coliform (6 hrs)	Flashpoint	1 Notify proper lab group immediately.
Hexavalent Chromium (24 Hrs)	TSS	2 Complete nonconformance memo.
BOD	Total Solids	
Nitrite	**TDS**	
Nitrate	Sulfide	
Ortho Phosphorus	Free Liquids	
Turbidity	Total Volatile Solids	
Surfactants	Aqueous Extractable Organics- ALL	
Sulfite	Unpreserved VOC's	
En Core Preservation	Ash	
Color		

Rev. 5/3/2001, Attachment to 1-REC-5.
Subject to QA Audit.

Reviewed by/date_____

Master Logbook Form

ate / r	Project Information: Client Information	Matrix	Batch #	Sample #	Biota	HNO3 (pl/gl): 125	250	500	1L	4L	H2SO4 (pl/gl): 125	250	500	1L	Unpreserved (pl/gl): encore	40	60	125	250	500	1L	HCL: 40	1L	MeOH: 60	NaOH: 250	500	ZnAc+ NaOH: 1L	Log'd by
	Submitter Name					✓	✓	✓	✓		✓	✓	✓	✓				✓	✓	✓	✓				✓	✓		
	Project Name																											
	Project #					✓	✓	✓	✓		✓	✓	✓	✓				✓	✓	✓	✓				✓	✓		
	Comments																											
																												QC
	Submitter Name																											
	Project Name																											
	Project #																											
	Comments																											
																												QC
	Submitter Name																											
	Project Name																		✓	✓	✓	✓				✓	✓	
	Project #																											
	Comments																											
																												QC
	Submitter Name																											
	Project Name																											
	Project #																											
	Comments																											
																												QC

Locations

A Archive Room
D&E Semi-volatiles (water)
F Semi volatiles (soil)
G Semi volatiles (extra)
S Subcontracted after receipt
V Volatiles
W Walk-in Sample Receiving
X Walk-in Wet Chemistry
Z Freezer

Page

Form I-4

LTS

Shelf	Batch & Sample Number(s)	Bottle type & Preservation	in	out	in	out	in	out	in	out	iu	out	in	Disposal Date & Haz Code	Return or Store

Comments: _____

_____

_____

Disposed

Storage Location_____

Project _____ Project #_____ Sample Type_____ Location_____

NONCONFORMANCE REPORT

NUMBER: _____
(QA ASSIGNED)

LABORATORY

INORGANIC: _____ DATE: _____

ORGANIC: _____ INITATOR _____

PROJECT NAME: _____ PROJECT NUMBER: _____

SAMPLE NUMBERS: _____ LIMS TEST GROUP ID: _____

DESCRIBE THE NONCONFORMANCE \ OCCURRENCE _____

ACTION TAKEN FOR THIS OCCURRENCE _____

GROUP LEADERS APPROVAL: _____ DATE: _____

QA MANAGER'S APPROVAL: _____ DATE: _____

CORRECTIVE ACTION FOLLOW-UP COMMENTS _____

COPY ROUTING

GROUP LEADER: _____ PROJECT COORDINATOR: _____

LAB MANAGER: _____ LAB DIRECTOR: _____

SAMPLE ENTRY NONCONFORMANCE MEMO (CLIENT NAME/CONTACT:

EnCHEM PROJECT NO: PROJECT NAME: PROJECT NUMBER:

Problem	Lab ID	Field ID	Comments/Corrective Action
No ice / Temperature			
Broken Bottles/ Cracked Cap			
Hold Time			
Headspace			
Labeling			
Volume			
Container			
Preservation			
Other			

APPENDIX J

SOP LISTING

SOP #	Title	Control		
	GENERAL (GEN)	Rev. #	DATE	Status
GEN-1	Preparation of Standard Operating Procedure	4	12/98	FINAL
GEN-2	Distribution, Receipt, Return, and Accessability of QA Documents	3	11/98	FINAL
GEN-3	Determination of Sample Holding Times	1	7/99	FINAL
GEN-4	Data Recording, Signatures, Dates, and Data Handling	7	3100	FINAL
GEN-5	Sample Disposal	1	11/97	Archive
GEN-6	Data Reduction, Review, Validation, and Reporting	1	5101	draft
GEN-7	Sample Chain of Custody	4	5/97	FINAL
GEN-8	Acknowledgement of Exposure to and Revision of Standard Operating Procedures	2	12/98	FINAL
GEN-9	Purchasing/ Supply Request/ Receiving	1	10/96	Archive
GEN-10	Courier Service	1	6/96	Archive
GEN-11	Closed			
GEN-12	Hazardous Waste Handling Procedures	0	11/97	Archive
GEN-13	Preparing Archive Boxes	1	11/00	FINAL
GEN-14	Saturday Sample Receipt	0	5198	FINAL
GEN-15	Nonconformance Memos	1	3100	FINAL
GEN-16	Laboratory Notebooks	1	3/00	FINAL
GEN-17	Software Installation/Validation	0	11198	FINAL
GEN-18	Training of Employees and Maintenance of Training Files	0	11/98	FINAL
GEN-19	Laboratory Contingency Plan for Sample Analysis	0	11198	FINAL
GEN-20	Data Review Checklists for Level 4 Data Packages	0	12/98	FINAL
GEN-21	Standard Laboratory Deliverable Formats	0	12/98	FINAL
GEN-22	Level 4 Data Package Assembly	0	12/98	FINAL
GEN-23	Policy for Data Confidentiality and Release of Data	0	11/98	FINAL
GEN-24	Label Removal	0	11/98	FINAL
GEN-25	Record keeping, Handling, and Disposition	0	11/98	FINAL
GEN-26	Identification of QC Samples on Laboratory Data	0	12/98	FINAL

SOP #	Title	Control		
	GENERAL (GEN)	Rev. #	DATE	Status
GEN-27	Project Management	0	1/99	FINAL
GEN-28	Review and Verification of Data Downloads	0	2'99	FINAL
GEN-29	Resolution of Client Complaints	0	3100	FINAL
GEN-30	Sample Acceptance Policy	0	4/00	FINAL
GEN-31	Sample Narratives	0	4/00	FINAL
GEN-32	Archive Database Entry/Query	0	5/00	FINAL
GEN-33	Hazardous Waste Contingency Plan	0	3/00	Archive
GEN-34	Archive Box Retrieval	0	5/00	FINAL
GEN-35	Special Requirements for Analysis	1	11/00	FINAL
GEN-36	Laboratory Waste Disposal	0	2/00	Draft

SOP #	Title	Control		
	LABORATORY (LAB)	Rev. #	DATE	Status
LAB-I	Pipettor Calibration	3	3/00	FINAL
LAB-2	Laboratory Corrective Action Form			**Archived**
LAB-3	Nonconformance Memos			**Archived**
LAB-4	Labeling of Reagents and Solutions			**Archived**
LAB-5	Recording of Solvent and Reagent Lot Numbers	1	8196	Final
LAB-6	Charting of Recoveries in Control Samples	3	12/98	Final
LAB-7	Corning 220 pH Meter Operation	2	11/00	Final
LAB-8	Traceability of Analytical Standards	2	2/97	Final
LAB-9	Acid Handling	1	4/00	Final
LAB-I 0	Thermom/Inst Temp Cal.	1	6/99	Final
LAB-I 1	Cornpositing and Homogenizing of Samples	1	4/00	Final
LAB-12	Refrigerator and Freezer Temperature Monitoring	4	11/97	Final
LAB-1 3	Drying Oven Temperature Monitoring	2	9/96	Final
LAB-14	MDL Determination	2	3/00	Final
LAB-15	DI Water System Check	3	11/98	Final
LAB-16	Soils, Total Solids	1	4100	Final
LAB-17	Monitoring and Changing Compressed Gas Cylinders	1	4/00	Final
LAB-I 8	Automatic Dishwasher Hobart Am-14	0	9/97	Final
LAB-19	Handling and Homogenization of Small Rodent Samples prior to Extraction and Digestion	0	10/99	Final
LAB-20	Orion pH Meter	1	4/00	Final
LAB-21	Preservation of VOC Samples in Solid Matricies by Method 5035	0	2/98	Final
LAB-22	Traceability of Laboratory Reagents	0	11/98	Final
LAB-23	ASTM- Soils, Total Solids.	1	4100	Final
LAB-24	Automatic Dishwasher	0	4/99	Final
LAB-25	Inorganics Glassware Cleaning	1	4100	Final
LAB-26	Toploader Balance Operation	1	4/00	Final
LAB-27	Preparation of Biological Tissues for Laboratory Determinations	0	6/99	Final

SOP #	Title	Control		
	LABORATORY (LAB)	Rev. #	DATE	Status
LAB-28	US Berkel Meat Chopper	0	8/00	Final
LAB-29	Waring Laboratory Blender	0	8/00	Final

SOP #	Title	Control		
	LEACHING PROCEDURES (LCH)	Rev. #	DATE	Status
LCH-1	Digital Hotplate/Stirrer PMC 730	0	7/99	FINAL
LCH-2	TCLP Leaching Procedure	2	1/01	FINAL
LCH3	TCLP Neutral Leach	0	7/99	FINAL
LCH-4	SPLP Leaching Procedure	1	1/01	FINAL
LCH-5	EPTox Leaching Procedure	0	7/99	FINAL
LCH-6	EPW (Water) Leach Indiana Leach Test	0	7/99	FINAL
LCH-7	ZHE Extraction Procedure for Volatile Compounds	0	7/99	FINAL
LCH-8	MEP Leaching Procedure	0	7/99	FINAL
LCH-9	ASTM Shake Extraction of Solid Waste With Water	1	4/00	FINAL
LCH-10	AFS Leaching Procedure	0	7/99	FINAL
LCH-11	ZHE Vessel Cleaning Procedure	0	7/99	FINAL

SOP #	Title	Control		
	METALS (MET)	Rev. #	DATE	Status
MET-1	Closed			
MET-2	Acid Digestion of Water for Dissolved or Total Recoverable Metals-ICP	8	2/00	FINAL
MET-3	Acid Digestion of Water for Total Metals-ICP	7	2/00	FINAL
MET4	Acid Digestion of Water for Total Metals-GFAA	6	2/00	FINAL
MET-5	Acid Digestion of Water for Arsenic and Selenium-GFAA	7	2/00	FINAL
MET-6	Acid Digestion of Solid Sample	9	10/00	FINAL
MET-7	Acid Digestion of Solid Samples for Silver, Antimony and Mercury	7	3/00	FINAL
MET-8	Acid Digestion of Solid Sample-GFAA			**Archived**
MET-9	Total Solids Dried at 103-105°C			**Archived**
MET-10	GFAA Spectraphotometer 51 OOZ (2)	3	4/00	FINAL
MET-l 1	AA Spectrophotometer P.E. 3100			**Archived**
MET-12	Open			
MET-l 3	Furnace Analysis of Lead	6	4/00	FINAL
MET-14	Furnace Analvsis of Selenium	5	7/99	FINAL
MET-l 5	Furnace Analysis of Antimony	3	4100	FINAL
MET-16	Furnace Analvsis of Thallium	5	4100	FINAL
MET-l 7	Open			
MET-l 8	Furnace Analysis of Silver	5	4/00	FINAL
MET-19	Analytical Calculations and Significant Figures			**Archived**
MET-20	Furnace Analvsis of Cadmium	7	4/00	FINAL
MET-21	Furnace Analysis of Arsenic	6	4/00	FINAL
MET-22	Furnace Analysis of Chromium	6	4/00	FINAL
MET-23	Furnace Analysis of Copper	3	4/00	FINAL
MET-24	Furnace Analysis of "Screen Samples"			**Archived**
MET-25	GFAA Spectrophotometer 51 OOZ (1)			**Archived**
MET-26	Closed			
MET-27	ICP-AES	10	3/01	FINAL
MET-28	ICP-P2			**Archived**
MET-29	Hardness by Calculation	3	4/00	FINAL
MET-30	Mercury Analysis of Aqueous Samples by CVAA	7	3/01	FINAL
MET-31	Mercury Analysis of Compositional Samples by CVAA	8	3/01	FINAL
MET-32	Digital Hotplate/Stirrer PMC 730			Trans. to LCH SOP
MET-33	TCLP Leaching Procedure			Trans. to LCH SOP
MET-34	TCLP Neutral Leach			Trans. to LCH SOP

SOP #	Title	Control		
	METALS (MET)	Rev. #	DATE	Status
MET-35	SPLP Leaching Procedure			Trans. to LCH SOP
MET-36	EPTox Leaching Procedure			Trans. to LCH SOP
MET-37	EPW (Water) Leach Indiana Leach Test			Trans. to LCH SOP
MET-38	ZHE Extraction Procedure for Volatile Compounds			Trans. to LCH SOP
MET-39	MEP Leaching Procedure			Trans. to LCH SOP
MET-40	ASTM Shake Extraction of Solid Waste With Water			Trans. to LCH SOP
MET-41	AFS Leaching Procedure			Trans. to LCH SOP
MET-42	TJA ICP Instrument Operating Procedure	3	7/00	FINAL
MET-43	Hexavalent Chromium Flame AA Analysis			**Archived**
MET-44	Hexavalent Chromium Chelation Extraction			**Archived**
MET-45	Microwave Digestion Aqueous	2	2/00	FINAL
MET-46	Microwave Digestion Compositional	2	2/00	FINAL
MET-47	Questron Q-wave 3000 Operation	2	2/00	FINAL
MET-48	ZHE Vessel Cleaning Procedure	0	1/97	Trans. to LCH SOP
MET-49	Hexavalent Chromium Colorimetric			**Archived**
MET-50	Graphite Furnace Atomic Absorption Spectrometer			**Archived**
MET-51	Acid Volatile Sulfide			**Archived**
MET-52	TJA ICAP 61 E			**Archived**
MET-53	Alkaline Extraction of Hexavalent Chromium			**Archived**
MET-54	GFAA Spectrophotometer 51 OOZ (3)			**Archived**
MET-55	Cation Exchange Capacity	0	7/00	FINAL
MET-56	Spiking Procedure for Metals Analysis	2	3/00	FINAL
MET-57	FIMS AA Spectrophotometer	2	3/00	FINAL
MET-58	Determination of Trace Metals by ICP-MS	4	8/00	FINAL
MET-59	Acid Digestion of Biota Samples	1	3/00	FINAL

SOP #	Title	Control		
	Quality Assurance (QAU)	Rev. #	Date	Status
QAU-1	Quality Assurance Facility Audit Procedures	2	12/98	Final

SOP #	Title	Control		
	RECEIVING	Rev. #	Date	Status
REC-1	LIMS Representation of Special Requests	0	5/94	**Archived**
REC-2	Sample Bottle Tags	1	9/97	Final
REC-3	Sample Bottle Requests and Preparation	1	9/96	Final
REC-4	Subcontracting	0	10/96	Final
REC-5	Cooler Receipt Log	1	12199	Final
REC6	Closed			
REC-7	Sample Receipt and Log-in	2	3100	Final
REC-8	Chain of Custody	1	10/96	Final
REC-9	Balance Calibration and Tare Wt. of 60-mL Containers	1	10/96	Final
REC-10	Sample Storage	1	8/96	Final
REC-11	EnCore Sampler Extrusion	0	9/96	Final
K EC-I 2	Closed			
REC-13	Balance Calibration and Initial Weighing for Total Solids	1	9/96	Final

SOP #	Title	Control		
	SEMIVOLATILES (SVO)	Rev. #	Date	Status
svo-1	Extraction of Water Samples for Base/Neutral/Acids	3	2/98	Final
svo-2	Extraction for TCLP Base/Neutral/Acids	3	2/98	Final
svo-3	Extraction of Water Samples for Base/Neutral/Acids Continuous Liquid-Liquid Extraction	4	12/98	Final
svo-4	Extraction of Soil Samples for Base/Neutral/Acids	3	2/98	Final
svo-5	Extraction of Soil Samples for Base/Neutral/Acids CLP Protocol	2	8/96	Final
SVO-6	Extraction of Water Samples for Organochlorine Pesticides/PC&	4	7/00	Final
SVO-7	Extraction for TCLP Organochlorine Pesticides	3	2/98	Final
SVO-8	Extraction of Water Samples for Organochlorine Pesticides/PCBs CLP Protocol	2	5/97	Final
svo-9	Extraction of Water Samples for Organophosphorus Pesticides	1	2194	Archived
SVO-10	Extraction of Soil Samples for Organochlorine Pesticides/PCBs	3	7/98	Final
svo-11	Extraction of Soil Samples for Organochlorine Pesticides/PCBs CLP Protocol	2	5/97	Final
SVO-12	Extraction of Soil Samples for Organophosphorus Pesticides	1	3/97	Archived
SVO-13	Extraction of Water Samples for Chlorinated Herbicides	3	4/00	Final
SVO-14	Extraction for TCLP Chlorinated Herbicides	2	3197	Final
SVO-15	Extraction of Water Samples for Total Petroleum Hydrocarbons as Diesel	1	2/94	Archived
SVO-16	Extraction of Water Samples for Diesel Range Organics (DRO)	2	6/95	Archived

SOP #	Title	Control		
	SEMIVOLATILES (SVO)	Rev. #	Date	Status
svo-17	Extraction of Soil Samples for Total Petroleum Hydrocarbons as Diesel	1	2/94	Archived
SVO-18	Extraction of Soil Samples for Diesel Range Organics (DRO)	2	6/95	Archived
svo-19	Medium Level Extraction of Soil Samples for Base/Neutral/Acids	2	5/97	Final
svo-20	Medium Level Extraction of Soil Samples for Base/Neutral/Acids CLP Protocol	2	5/97	Final
SVO-21	Medium Level Extraction of Soil Samples for Toxaphene and PCBs	2	5/97	Final
SVO-22	Extraction of Oil Samples for PCBs	3	3/00	Final
SVO-23	Extraction of Water Samples for Total Petroleum Hydrocarbons by Infrared Spectroscopy	1	2/97	Final
SVO-24	Extraction of Soil Samples for Total Petroleum Hydrocarbons by Infrared Spectroscopy	2	2/97	Final
SVO-25	Florisil Cleanup	2	3/97	Final
SVO-26	Gel Permeation Chromotography	2	11/97	Final
SVO-27	Sulfur Cleanup	1	9/96	Final
SVO-28	Sulfuric Acid Cleanup	2	8/96	Final
SVO-29	Total Petroleum Hydrocarbons as Diesel (Cal. Method.)	1	2/94	Archived
SVO-30	Diesel Range Organics	2	2/94	Archived
SVO-31	Analysis of Organophosphorus Pesticides	0	2/94	Archived
SVO-32	Analysis of Chlorinated Herbicides	2	10/97	Final
SVO-33	Analysis of Toxaphene	3	6/99	Final
SVO-34	Analysis of PCBs	1	2/94	Archived
SVO-35	Analysis of Organochlorine Pesticides and PCBs	4	12/97	Final
SVO-36	Analysis of Organochlorine Pesticides and PCBs CLP Protocol	2	8/96	Final
SVO-37	Analysis of BNA Compounds by GC/MS	8	4/01	Final
SVO-38	Analysis of BNA Compounds	0	2/94	Final

SOP #	Title	Control		
	SEMIVOLATILES (SVO)	Rev. #	Date	Status
	by GC/MS CLP Protocol			
svo-39	Extraction of Soil Samples for PAHs by HPLC	0	5/96	Final
SVO-40	Extraction of Water Samples for PAHs by HPLC	0	5/96	Final
svo-41	Analysis of Polynuclear Aromatic Hydrocarbons (PAH)	2	12/97	Final
svo-42	Herbicides in Soil Extraction	1	5/00	Final
SVO-43	Glassware Cleaning	2	12/98	Final
svo-44	Prevention of Cross Contamination and Prescreening of Solvents	0	3/97	Final
svo-45	Extraction of PCB Wipe Samples	1	4/00	Final
SVO-46	Benzene Analysis by FID			Archived
svo-47	Analysis of Water Samples for Methanol	0	9/96	Final
SVO-48	Open			
svo-49	Analysis of PAH Method 610	2	6/00	Final
svo-50	Determination of Nitroglycerin in Soil/Sediment by HPLC	0	8/97	Final
svo-51	Analysis of Organochlorine Pesticides by GC	5	6/00	Final
SVO-52	Analysis of Polychlorinated Biphenyls (PCBs) by GC	3	1/00	Final
SVO-53	Sonic Probe Use and Maint.	1	6/99	Final
svo-54	Extraction of Neutrogena Coal Tar Based Samples for PAH.	1	3/99	Final
svo-55	Extraction of Neutrogena Coal Tar Based Samples for Benzo(a)Pyrene.	0	3199	Final
SVO-56	ASE	0	7/99	Final
svo-57	Florisil Column Cleanup for PCB's	0	4/99	Final
svo-58	Silica Gel Cleanup and Separation of Pesticides and PCB's.	0	4/99	Final
svo-59	Determination of Lipids in Tissues, Fats, and Plants.	0	4199	Final
SVO-60	Extraction of Biological Samples for Organochlorine Pesticides/PCB's	1	7100	Final

SOP #	Title	Control		
	SEMIVOLATILES (SVO)	Rev. #	Date	Status
SVO-61	Extraction of Biological Samples for PAH's	1	2/00	Final
SVO-62	Congener specific analysis of PCB in Tissue	1	10/99	Final
SVO-63	Analysis of PAH in Soil, Water and Biota by GCJMS-SIM	2	12100	Final
svo-64	Congener specific analysis of PCB in Soils and Sediments	1	10/99	Final
SVO-65	Congener specific analysis of PCB in Water,	0	*2J00*	Final
svo-66	Extraction of Biological Samples for PAH's by GCJMS-SIM	0	4/01	Final
SVO-67	Extraction of Soil Samples for PAH's by GCJMS-SIM	2	4/01	Final
svo-68	Determination of N-Methyl Carbamates and N-Methyl Carbamoyl Oximes in Water	0	*5J00*	Final
SVO-69	Nitroaromatics and Nitramines in Soil/Sediments by HPLC	0	6/00	Final
svo-70	Extraction of Biota Samples for Base/Neutral/Acids	0	*7J00*	Final
svo-7 1	Nitroaromatics and Nitramines in Water by HPLC	0	11/00	Final
SVO-72	Generation of Diazomethane from Diazald	0	8/00	Final
svo-73	Analysis of Semivolatile (BNA) Compounds by GCJMS in Biological Matrices	0	8/00	Final
svo-74	Extraction of Water Samples for Pentachlorophenol by GUMS-SIM	0	11/00	Final
svo-75	Analysis of Pentachlorophenol in Water by GCJMS-SIM HP-5973	0	11/00	Final
SVO-76	Extraction of Water Samples for PAH's by GUMS-SIM	0	4101	Final

SOP #	Title	Control		
	Volatile Organics (VOA)	Rev. #	Date	Status
VOA-1	Storage Blank Preparation and Analysis	0	8/97	Final
VOA-2	Closed			
VOA-3	Prep. of VOA Stock and Working Standards	1	3/95	Archived
VOA-4	Analysis of Volatile Organics by ELCD/PID GC	1	8/94	Archived
VOA-5	Volatile Organics Analysis by GC/MS 8260	6	11/98	Final
VOA-6	Low Level VOA by *GCJMS* Method 524.2	1	8/94	Final
VOA-7	VOA GC/MS Report Submission	1	2195	Archived
VOA-8	IOP for VOA *GCJMS*	1	3/95	Final
VOA-9	Volatile Organics Analysis of Solid Matricies Methods 5035/8260B	1	11/98	Final
VOA-10	Glassware Cleaning Procedure	0	7/98	Final
VOA-11	Volatile Organics Analysis by GC/MS Method 624	0	6/00	Final

SOP #	Title	Control		
	WET CHEMISTRY (WCM)	Rev. #	DATE	Status
WCM-1	Total Suspended Solids Dried at 103-l 05°C Volatile Solids Ignited at 550°C	8	1/01	FINAL
WCM-2	Total Organic Carbon (TOC) in Water	9	4/99	FINAL
WCM-3	Phenolics, Total Recoverable Distillation	3	4/00	FINAL
WCM-4	Oil & Grease, Total Recoverable, Gravimetric Separatory Funnel Extraction	2	8/99	FINAL
WCM-5	Chemical Oxygen Demand Open Reflux Method Mid Level	4	8/96	Archived
WCM-6	OPEN			
WCM-7	Nitrogen, Ammonia, Colorimetric, Distillation	2	3/94	**Archived 10/97**
WCM-8	Phenolics, Total Recoverable Automated Analysis	7	1/01	FINAL
WCM-9	Total Organic Carbon Analysis in Soils, Sludge, and Solid Waste	11	1/01	FINAL
WCM-10	Total Solids Dried at 103-105°C; Volatile Solids Ignited at 550°C	4	4/00	FINAL
WCM-11	Total Dissolved Solids Dried at 180°C; Volatile Solids Ignited at 550°C	4	8/99	FINAL
WCM-12	Alkalinity, Titrimetric, pH 4.5	5	8/96	**Archived**
WCM-13	Alkalinity, Titrimetric, pH 8.3 & 4.5	5	10/00	FINAL
WCM-14	Iodide Analysis, Titrimetric	2	8/96	**Archived 12/97**
WCM-15	Bromide Analysis, Titrimetric	1	8/96	**Arc hived 12/97**
WCM-16	Acidity, Titrimetric	2	8/99	FINAL
WCM-17	Conductivity	5	8/99	FINAL
WCM-18	TOC Analyzer	5	4/99	FINAL
WCM-19	Chloride, Automated Analysis	7	1/01	FINAL
WCM-20	Sulfate	6	1/01	FINAL
WCM-21	Sulfide-Water	4	7/99	FINAL
WCM-22	Sulfides Titrimetric-Soil	4	7/99	FINAL
WCM-23	Total Cyanide, Automated Analysis	7	11/00	FINAL
WCM-24	Turbidity	3	8199	FINAL
WCM-25	Nitrogen, Ammonia, Distillation Procedure	6	1/01	FINAL
WCM-26	TKN, Colorimetric	2	8/96	**Archived 12/97**

SOP #	Title	Control		
	WET CHEMISTRY (WCM)	Rev. #	DATE	Status
WCM-27	UV-Vis Single Beam Spectrophotometer	2	8/96	Archived 12/97
WCM-28	Total Alkalinity Automated Analysis	6	1/01	FINAL
WCM-29	Automated Ion Analyzer System	5	3J00	FINAL
WCM-30	Cyanide Distillation Aqueous and Compositional	1	9/97	Archived 10197
WCM-31	Reactive Cyanide and Sulfide, Preparation	4	4/00	FINAL
WCM-32	Chemical Oxygen Demand Open Reflux Method Low Level	4	8/96	Archived
WCM-33	Nitrate-Nitrite Automated Analysis	7	1/01	FINAL
WCM-34	Cyanide, Weak and Dissociable Distillation	1	7/99	FINAL
WCM-35	Cyanide Amenable to Chlorination- Distillation	2	7/99	FINAL
WCM-36	Nitrogen- Nitrite Automated Analysis	4	1/01	FINAL
WCM-37	Midi-Dist Cyanide Distillation	4	11/00	FINAL
WCM-38	Color	1	8/99	FINAL
WCM-39	COD - Colorimetric Manual (Vial Low)	5	10/00	FINAL
WCM-40	COD - Colorimetric Manual (Vial Mid)	4	4100	FINAL
WCM-41	Oil and Grease, Total Recoverable, Compositional	4	3J00	FINAL
WCM-42	Fluoride by Ion Selective Electrode - Aqueous	8	8/99	FINAL
WCM-43	Fluoride by Ion Selective Electrode - Soils	4	8/99	FINAL
WCM-44	Phosphorus. All Forms Automated Analysis	5	3/00	FINAL
WCM-45	Flashpoint	2	8/99	FINAL
WCM-46	Paint Filter Liquids Test	2	8/99	FINAL
WCM-47	Specific Gravity	2	3J00	FINAL
WCM-48	Ferrous Iron	2	1/01	FINAL
WCM-49	Nitrogen Ammonia Ion Selective Electrode	1	8/99	FINAL
WCM-50	Bromide, Automated Analysis	1	8/99	FINAL
WCM-51	Biochemical Oxygen Demand	1	1/01	FINAL
WCM-52	Total Kjeldahl Nitrogen, I.S.E.	1	7/99	Archived
WCM-53	Turbidimetric Sulfate	1	8/99	FINAL
WCM-54	pH Analysis	1	8/99	FINAL
WCM-55	Settleable Solids	1	8/99	FINAL
WCM-56	Oxidation Reduction Potential- Th	1	8/99	FINAL

SOP #	Title	Control		
	WET CHEMISTRY (WCM)	Rev. #	DATE	Status
WCM-57	Free Aciditv. Titrimetric	1	8/99	FINAL
WCM-58	Ammonia Nitrogen- Automated Analysis	4	1/01	FINAL
WCM-59	Ion Chromatography - IOP	2	3/00	FINAL
WCM-60	Anions by ion Chromatography	5	1/01	FINAL
WCM-61	Chromium, Hexavalent- Colorimetric	1	1/01	FINAL
WCM-62	Alkaline Extraction of Hexavalent Chromium	1	12/99	FINAL
WCM-63	AVS and SEM in Sediment	1	4/00	FINAL
WCM-64	Total Kieldahl Nitroaen	2	1/01	FINAL
WCM-65	Thiocyanate Determination	2	10/00	FINAL
WCM-66	Particle Size Reduction	0	9/00	FINAL
WCM-67	Oil & Grease, SPE	0		Draft
WCM-68	O&G SPE-DEX 4790 System	0		Draft
WCM-69	Appolo 9000 IOP	0		Draft
WCM-70	TOC Water by Appolo 9000	0		Draft

APPENDIX K

METHOD REFERENCES

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2. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act," CFR Part 136, October 26, 1984.

3. "Methods for Chemical Analysis of Water and Wastes," EPA 600/4-79-020, U.S. Environmental Protection Agency, March 1979.

4. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, USEPA, (3rd Edition).

5. Standard Methods for the Examination of Water and Wastewater, 18th ed., American Public Health Association.

6. "The Analysis of Aromatic Chemicals in Water by the Purge and Trap Method," Method 503.1, U.S. Environmental Protection Agency, Physical and Chemical Methods Branch, Cincinnati, Ohio, May 1980.

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9. "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol, and Pesticides in Water and Wastewater," Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio, September 1978.

10. "Annual Book of ASTM Standards, Sections 11 .01 and 11.02, Water and Environmental Technology," American Society for Testing and Materials.

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13. "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue," Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, USEPA, Cincinnati, Ohio, (April 1981).

14. Manual of Analytical Methods for Analysis of Pesticides in Human and Environmental Samples, USEPA, EPA 600/8-80-038 (June).

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