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FINAL

Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant (NIROP) Fridley, Minnesota

Prepared For

Naval Facilities Engineering Command
Southern Division
North Charleston, South Carolina

Prepared By

PARSONS ENGINEERING SCIENCE, INC.
1700 Broadway, Suite 900 • Denver, Colorado 80290

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FINAL
WORK PLAN FOR FIELD APPLICATION TO ENHANCE *IN-SITU* BIOREMEDIATION OF CHLORINATED SOLVENTS VIA VEGETABLE OIL INJECTION AT THE NAVAL INDUSTRIAL RESERVE ORDNANCE PLANT (NIROP), FRIDLEY, MINNESOTA

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Prepared for:

NAVAL FACILITIES ENGINEERING COMMAND
SOUTHERN DIVISION
NORTH CHARLESTON, SOUTH CAROLINA

Prepared by:

Parsons Engineering Science, Inc.
1700 Broadway, Suite 900
Denver, Colorado 80290

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ACRONYMS AND ABBREVIATIONS

θC	degrees Celsius
μg/L	micrograms per liter
ACP	Anoka County Park
BCT	BRAC Cleanup Team
BEHP	bis(2-ethylhexyl)phthalate
Bgs	below ground surface
BRAC	Base Closure and Realignment Act
CAHs	chlorinated aliphatic hydrocarbons
COCs	contaminants of concern
DCA	dichloroethane
DCE	dichloroethene
DO	dissolved oxygen
DOT	Department of Transportation
DPT	direct-push technology
DQO	Data Quality Objective
EnSafe	EnSafe, Inc.
ft^2/day	square feet per day
ft/day	feet per day
ft/ft	foot per foot
ft/year	feet per year
gpm	gallons per minute
HSWA	Hazardous and Solid Waste Amendments
ID	inside-diameter
IDW	investigation-derived waste
IRP	Installation Restoration Program
K	hydraulic conductivity
lb/in^2	pounds per square inch
LCSs	laboratory control samples
LMBs	laboratory method blanks
LTM	long-term monitoring
MCLs	maximum contaminant levels
MDH	Minnesota Department of Health
mg/L	milligrams per liter
MPCA	Minnesota Pollution Control Agency
MSDS	Material Safety Data Sheet
N	nitrogen
NAPL	non-aqueous phase liquid
NAS	Naval Air Station
NAVFAC	Naval Facility Engineering Command
NSA	Naval Support Activity
ORC	oxygen releasing compound
ORP	oxidation-reduction potential
Parsons ES	Parsons Engineering Science, Inc.
PCE	tetrachloroethene
POC	point of contact
PVC	polyvinyl chloride
QA/QC	quality assurance/quality control
QAPP	Quality Assurance Project Plan
RBCs	risk-based concentrations
RCRA	Resource Conservation and Recovery Act
redox	reduction-oxidation

RFI	RCRA Facility Investigation
ROD	Record of Decision
SSL	soil screening levels
SWMU	Solid Waste Management Unit
SVOCs	semi-volatile organic compounds
T	transmissivity
TCA	trichloroethane
TCE	trichloroethene
TOC	total organic carbon
UDLP	United Defense Limited Partnership
UIC	underground injection control
USEPA	United States Environmental Protection Agency
VC	vinyl chloride
VOCs	volatile organic compounds

SECTION 1

INTRODUCTION

This work plan presents the scope of work required to conduct a field application for enhanced *in-situ* bioremediation of chlorinated solvents via vegetable oil injection in Anoka County Park (ACP) near the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota. This work plan has been prepared for Southern Division, Naval Facilities Engineering Command by Parsons Engineering Science, Inc. (Parsons ES).

This work plan was developed based on review of the 1999 Annual Monitoring Report, Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota (Tetra Tech NUS, Inc., 2000a) and the Field Investigation Report at the NIROP and Anoka County Riverfront Park, Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota (Tetra Tech NUS, 2000b) and discussions between Parsons ES, CH2M Hill Constructors, Inc., the United States Environmental Protection Agency (USEPA), the Minnesota Pollution Control Agency (MPCA), Anoka County Parks, TechLaw, Tetra Tech NUS, the Minnesota Department of Health (MDH), and Southern Division, Naval Facilities Engineering Command.

1.1 SCOPE OF WORK

This project is being conducted by Parsons ES and Southern Division, Naval Facilities Engineering Command to document the enhanced bioremediation of chlorinated solvents dissolved in groundwater and sorbed to the aquifer matrix by injecting vegetable oil into the subsurface below the water table. Specifically, the objective of this field application is to determine if vegetable oil injection is a viable treatment option for volatile organic compounds (VOCs) in groundwater at the NIROP facility and adjacent areas including the Anoka County Park. The approximate location of the vegetable oil injection test is shown on Figure 1.1. Site-specific activities in support of the enhanced bioremediation field application will include:

- Installation of 1 new background monitoring well in the vicinity of existing monitoring well MS-46S, 3 vegetable oil injection wells, 9 groundwater monitoring wells, and 3 contingency monitoring wells;

- Baseline (i.e., pre-injection) sampling of groundwater at the newly installed injection wells, groundwater monitoring wells, contingency monitoring wells, and existing wells MS-46S, 27S, and MS-47S in accordance with the *Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater* (USEPA, 1998);

Figure 1.1 Location of Vegetable Oil Injection Pilot Test

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- ξ Pre-injection aquifer testing (hydraulic conductivity) of the 3 injection wells and 6 monitoring wells;
- ξ Plumbing of the injection wells and injection of up to 3,600 gallons of food-grade vegetable oil (1,200 gallons per well);
- ξ Post-injection aquifer testing (hydraulic conductivity) of the previously-tested 3 injection wells;
- ξ Surveying of the newly installed injection and monitoring wells;
- ξ Post-injection sampling of groundwater and vegetable oil (if present) at the newly installed monitoring and injection wells, in accordance with the *Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater* (USEPA, 1998) at 2, 5, 8, and 12 months after injection; and
- ξ Preparation of a report detailing the results of the field application.

The materials and methodologies to accomplish these activities are described in Section 4.

This work plan consists of eight sections, including this introduction (Section 1), and four appendices. Section 2 presents a review and analysis of available, previously reported, site-specific data. Section 3 provides an overview of biodegradation of chlorinated solvents. Section 4 describes system installation and the procedures to be followed for data collection. Section 5 describes the quality assurance/quality control (QA/QC) measures to be used during this project. Section 6 discusses a contingency plan. Section 7 describes the data analysis report, and Section 8 contains the references used in preparing this document. Appendix A contains an addendum to the NIROP Fridley Project Health and Safety Plan (CH2M Hill Constructors, Inc. 2000b) which will address health and safety aspects particular to vegetable oil injection activities. Appendix B contains a listing of containers, preservatives, packaging, and shipping requirements for soil and groundwater analytical samples. Appendix C lists examples of field forms to be used during the field work. Appendix D contains the USEPA Region 5-approved Quality Assurance Project Plan (QAPP) developed to address the data needs of this project. Appendix E contains the laboratory Quality Assurance Plan that will be used during analysis of samples collected during this program (En Chem, 2001). Appendix F contains standard operating procedures that will be in effect when samples collected during this program are analyzed by Enchem, Inc. Appendix G contains the MPCA-approved sampling standard operating procedure.

1.2 BACKGROUND

The NIROP Facility and ACP is located in Fridley, Minnesota in the southern most tip of Anoka County, just east of the Mississippi River. In September 1990 the Record of Decision (ROD) for the NIROP facility was signed by representatives of the U.S. Navy, USEPA Region 5, and the MPCA. The ROD specified hydraulic containment and recovery of all future migration of groundwater from the NIROP facility and the recovery, to the extent feasible, of contamination downgradient of the NIROP facility. To achieve this, groundwater extraction wells were installed. The initial extraction system began

operating in September 1992. Two additional extraction wells were constructed and placed into operation in June 1995. Another upgrade of the extraction system is expected in May 2001. Treatment of extracted groundwater involved a two-phased plan: Phase 1 involved discharging groundwater from the extraction system to an existing sanitary sewer with treatment provided at a local wastewater treatment facility; Phase II involved construction and operation of a groundwater treatment facility to provide long-term groundwater treatment. Groundwater treated by the Phase II treatment system is being discharged to the Mississippi River via a National Pollutant Discharge Elimination System (NPDES)/Metropolitan Council Environmental Services (MCES) State Disposal System (SDS) storm sewer discharge permit. The Phase II treatment system began operating in December 1998. Under Phase II, the combined discharge from the extraction system is fed through a feed system and air stripping units for treatment before the effluent is discharged to the Mississippi River through outfall 020 (NPDES/SDS Permit MN0000710).

SECTION 2

SITE-SPECIFIC DATA REVIEW

Site-specific data were reviewed to evaluate groundwater flow and contaminant transport and to determine locations for the installation of injection and groundwater monitoring wells. Section 2.1 presents a synopsis of hydrogeologic data made available to Parsons ES. Section 2.2 presents the distribution of contamination at the site.

The following sections are based upon a review and summary of data from the following sources:

- *1999 Annual Monitoring Report, Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota* (Tetra Tech NUS, Inc., 2000a);
- April 2000 *Field Investigation Report at the NIROP and Anoka County Riverfront Park, Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota* (Tetra Tech NUS, Inc., 2000b);
- *Technical Memorandum to the 1999 Annual Monitoring Report and Field Investigation Report, Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota* (Tetra Tech NUS, Inc., 2001).

The reader is referred to these documents for a more detailed review of site-specific data. Additional sources are referenced within the text. Based on review of available data, the pilot test will be conducted in the vicinity of well MS-46S located in ACP. Because of this, much of the discussion in the section focuses on the area around this well.

2.1 HYDROGEOLOGY

This section provides a brief discussion of the hydrogeology and hydraulic monitoring data at the NIROP facility and ACP in the vicinity of well MS-46S. A summary of the local hydrogeology at the facility is discussed including a description of the groundwater flow and the interaction between the aquifer system and the adjacent Mississippi River.

The hydrogeology within the vicinity of the NIROP facility is consistent with the regional hydrogeology where four aquifers or aquifer zones govern groundwater movement through the subsurface. These aquifers are, from shallow to deep, the unconsolidated Quaternary aquifer zone, the Prairie du Chien/Jordan aquifer (PC), the Franconia/Ironton/Galesville (FIG) aquifer, and the Mount Simon/Hinckley/Fond du Lac (MHF) aquifer. The relationship between the unconsolidated aquifer zone and the PC aquifer are illustrated in Table 2.1. More detailed descriptions can be found in the OU3 RI (Tetra Tech NUS, 1999) and the Field Investigation Report (Tetra Tech NUS, 2000b).

Table 2.1 Stratigraphic Information

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Because the vegetable oil injection pilot test will be conducted near well MS-46S in the shallow interval of the Quaternary aquifer system, the remainder of this section will focus on this stratigraphic interval.

The unconsolidated Quaternary aquifer zone consists of glacial drift (i.e., glacial outwash and till deposits) overlain by glacial-fluvial deposits. It is referred to as an aquifer zone because it consists of two poorly defined aquifers, a shallow unconfined aquifer (shallow drift aquifer) and a deep confined or semi-confined aquifer (deep drift aquifer). These aquifers consist of sand and gravel and they are separated by a silt or clay layer that ranges in thickness from 0 to 47 feet in the NIROP area. These two aquifers vary significantly in thickness and hydraulic connection throughout the site. The shallow unconfined aquifer ranges in thickness from 41 to 93 feet, and the deep confined or semi-confined aquifer ranges in thickness from 19 to 55 feet across the site. Although monitoring wells at the site have been designated as shallow and intermediate there appears to be no or very little hydraulic separation between these monitored intervals. Hence, both of these zones make up the shallow unconfined aquifer at the site. Cross-Section A-A' (Figure 2.1) illustrates the stratigraphic relationship of the shallow drift and deep drift aquifers in the pilot-study area.

2.1.1 Groundwater Flow Under Pumping Conditions

Under current pumping conditions the general horizontal groundwater flow direction across the NIROP site in the Quaternary aquifer zone is generally toward the southwest. Groundwater from the Quaternary aquifer zone discharges into the Mississippi River and underlying alluvium.

Groundwater flow in the far southwestern portion of the NIROP site (i.e., south end of ACP and western portion of the UDLP) does not follow these general trends in the Quaternary aquifer zone. A groundwater ridge is present near well MS-46S in ACP as shown on the potentiometric surface contour map for pumping conditions (November 30, 1999) for the shallow Quaternary aquifer (Figure 2.2). As a result of this ridge, groundwater flows in a radially outward pattern in the ACP.

2.1.2 Non-Pumping Conditions and Effect of Extraction Wells

The potentiometric surface contour map for non-pumping conditions (January 4, 2000) for the shallow Quaternary aquifers is presented in Figure 2.3. The entire extraction system was shutdown December 23, 1999. The aquifer system was allowed to recover for 13 days to reach static non-pumping water level conditions. Under static (i.e., non-pumping) conditions, the groundwater flow direction across the site is generally the same for the unconsolidated aquifers and the underlying PC bedrock aquifer as under pumping conditions.

As expected, water levels in the extraction wells recovered significantly (as much as 36.9 feet in well AT- 5B). The water level recovery in monitoring wells was much less ranging from 0.30 to 4.59 feet, 0.54 to 5.31 feet, and 1.69 to 5.73 feet in the shallow, intermediate and deep monitored intervals. A visual comparison of the head differences under pumping and non-pumping conditions (Figures 2.2 and Figure 2.3) reveals that the

Figure 2.1 Cross-Section A-A'

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Figure 2.2 Potentiometric Surface Map of Pumping Conditions

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Figure 2.3 Potentiometric Surface Map of Non-Pumping Conditions

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groundwater ridge remained in the ACP under static aquifer conditions indicating that the ridge is a result of natural conditions and is only accentuated by the groundwater extraction system.

2.1.3 Interaction Between the Aquifer System and the Mississippi River

The Mississippi River borders Anoka County Park to the west. It is expected that the Mississippi River and the underlying alluvium are hydraulically connected. The alluvium consists of sand and gravel (Schoenberg, 1994) and is estimated to be as much as 60 feet thick under the river (Lindgren 1990). To evaluate the interaction between the unconsolidated aquifer system and the Mississippi River, pressure transducers and data loggers were installed to record the rise and fall of the water levels in the aquifers in relation to the rise and fall of the river stage of the Mississippi River (Tetra Tech NUS, 2000b).

The hydraulic heads in the aquifer were found to be higher in all wells within 500 feet of the river compared to the river stage even during rapid changes in the river level. This head difference indicates that throughout the entire year (i.e., during all seasons) water in the aquifer system flows towards and discharges into the Mississippi River. This indicates that there were no bank storage effects, even at high river stages, during the data collection period. Thus, the Mississippi River is a gaining stream at all times of the year. (Tetra Tech NUS, 2000b).

2.2 GROUNDWATER QUALITY

Groundwater samples were collected and analyzed in the Spring (April 19-22) and Fall (November 2-5) of 1999. The results of these sampling events are described in more detail in the 1999 Annual Monitoring Report (Tetra Tech NUS, 2000a). Consistent with historical observations, TCE is the most extensive contaminant encountered in groundwater at the NIROP and adjacent ACP site. TCE was detected in 99 of 101 wells sampled in the fall 1999 sampling event. Detected concentrations of TCE ranged from 0.59 to 18,000 Πg/L in the fall 1999 sampling round. Dissolved TCE is present at generally higher concentrations in the unconfined aquifer (concentrations range from 0.59 to 18,000 Πg/L in the shallow and intermediate monitoring intervals) than the deep semi-confined aquifer (concentrations range from 1.8 to 5,200 Πg/L) (Tetra Tech NUS, 2000a). TCE in the PC aquifer is significantly lower than in shallow intervals (concentrations ranged from non-detect to 7.9 Πg/L in 1999) (Tetra Tech NUS, 2000a). Generally, TCE in groundwater extends from north of the NIROP's northern boundary to the southwest, along the direction of groundwater flow to the Mississippi River in the shallow, intermediate, and deep monitored intervals.

Based upon the results of the NIROP/ACP field investigation results (Tetra Tech NUS, 2000a, 2000b) TCE is present at elevated levels in the shallow aquifer in the southwestern portion of the NIROP and in ACP. A plan view isoconcentration map of the spatial distribution of TCE contamination in the shallow groundwater interval in the pilot study area near MS-46S is shown on Figure 2.4, based on groundwater samples collected in the fall of 1999. The concentrations are significantly lower northeast of East River Road (ranging from 170 to 730 Πg/L) than the concentrations present in the ACP (42 to 18,000 Πg/L) (See Figure 4-19 of Tetra Tech NUS, 2000a). More specifically, the concentrations in the central portion of the ACP are locally elevated ranging from 1,100

Figure 2.4 Trichloroethene (TCE) Concentrations

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to 18,000 µg/L at well MS-46S. This zone of elevated concentrations generally corresponds with the location of the groundwater ridge (Figure 2.2) which may represent a stagnation zone. This groundwater stagnation zone may allow elevated concentrations to exist in this area. The primary reason for implementing the pilot scale vegetable oil injection test near well MS-46S is because this well exhibited the highest TCE concentration in the Fall of 1999.

SECTION 3

ENHANCED BIOREMEDIATION OF CHLORINATED SOLVENTS USING VEGETABLE OIL

3.1 BIODEGRADATION OF CHLORINATED SOLVENTS

Chlorinated solvents can be transformed, directly or indirectly, by biological processes (e.g., Bouwer *et al.*, 1981; Miller and Guengerich, 1982; Reineke and Knackmuss, 1984; Wilson and Wilson, 1985; de Bont *et al.*, 1986; Nelson *et al.*, 1986; Spain and Nishino, 1987; Bouwer and Wright, 1988; Little *et al.*, 1988; Mayer *et al.*, 1988; Arciero *et al.*, 1989; Cline and Delfino, 1989; Freedman and Gossett, 1989; Folsom *et al.*, 1990; Harker and Kim, 1990; Alvarez-Cohen and McCarty, 1991a, 1991b; DeStefano *et al.*, 1991; Henry, 1991; Sander *et al.*, 1991; McCarty *et al.*, 1992; Hartmans and de Bont, 1992; Chapelle, 1993; McCarty and Semprini, 1994; Vogel, 1994; Suflita and Townsend, 1995; Bradley and Chapelle, 1996; Bradley and Chapelle, 1998; Spain, 1996). Biodegradation of chlorinated solvents, also termed chlorinated aliphatic hydrocarbons (CAHs), and chlorinated benzenes results from the same general processes that bring about biodegradation of fuel hydrocarbons. However, a more complex series of processes often is involved, as CAHs and chlorinated benzenes may act either as substrates (electron donors) or electron acceptors depending upon the prevailing geochemical conditions.

Chlorinated solvents may undergo biodegradation through three different pathways: (1) use as an electron acceptor, (2) use as an electron donor, or (3) cometabolism, which is degradation resulting from exposure to a catalytic enzyme fortuitously produced during an unrelated process. At a given site, one or all of these processes may be operating, although at many sites the use of CAHs as electron acceptors appears to be the most likely.

In a pristine aquifer, native organic carbon is utilized as an electron donor and dissolved oxygen (DO) is utilized first as the prime electron acceptor. Where anthropogenic carbon (e.g., vegetable oil, fuel hydrocarbons, landfill leachate, less-chlorinated CAHs, or chlorinated benzenes with four or fewer chlorines) is present, it also may be utilized as an electron donor. Most chlorinated solvents that can act as electron donors have thus far only been demonstrated to do so under aerobic conditions, with the notable exception of vinyl chloride (VC) (Bradley and Chapelle, 1996 and 1998). After the DO is consumed, anaerobic microorganisms typically use native electron acceptors (as available) in the following order of preference: nitrate, manganese oxide, ferric iron oxyhydroxide, sulfate, and finally carbon dioxide. Evaluation of the distribution of these electron acceptors can provide evidence of where and how biodegradation of chlorinated solvents is occurring.

Under anaerobic conditions, biodegradation of chlorinated solvents usually proceeds through a process called reductive dehalogenation. This is the only biological reaction known to degrade PCE, TCE, TCA, carbon tetrachloride, and chlorinated benzenes with more than four chlorines. During reductive dehalogenation, the halogenated hydrocarbon is used as an electron acceptor, not as a source of carbon, and a halogen atom is removed and replaced with a hydrogen atom. Figure 3.1 illustrates the transformation of chlorinated ethenes via reductive dehalogenation. In general, reductive dehalogenation occurs by sequential dechlorination. For the chlorinated ethenes, dechlorination progresses from PCE to TCE to DCE to VC to ethene.

Likewise, for the chlorinated methanes, dechlorination progresses from carbon tetrachloride to chloroform to methylene chloride to chloromethane to methane. Depending upon environmental conditions, these dechlorination sequences may be interrupted, with other processes then acting upon the products. During reductive dehalogenation of TCE, all three isomers of DCE theoretically can be produced; however, Bouwer (1994) reports that under the influence of biodegradation, *cis*-1,2-DCE is a more common intermediate than *trans*-1,2-DCE, and that 1,1-DCE is the least prevalent intermediate of the three DCE isomers. Reductive dehalogenation of chlorinated solvent compounds is associated with the accumulation of daughter products and an increase in chloride.

Reductive dehalogenation affects each of the chlorinated solvents differently. PCE, TCE, and carbon tetrachloride are the most susceptible of these compounds to reductive dehalogenation because they are the most oxidized. Conversely, VC and chloromethane are the least susceptible to reductive dehalogenation because they are the least oxidized of these compounds. Reductive dehalogenation has been demonstrated under nitrate- and sulfate-reducing conditions, but the most rapid biodegradation rates, affecting the widest range of CAHs, occur under methanogenic conditions (Bouwer, 1994).

Because CAH compounds are used as electron acceptors, there must be an appropriate source of carbon for microbial growth in order for reductive dehalogenation to occur. Potential carbon sources include vegetable oil, fuel hydrocarbons, low-molecular-weight compounds (e.g., lactate, acetate, methanol, or glucose) present in natural organic matter or less-chlorinated compounds such as VC, DCE, chloroethane, or DCA. An evaluation of chlorinated ethene groundwater data suggests that the natural biodegradation of chlorinated ethenes near the NIROP facility is electron-donor limited. Innocuous, food-grade vegetable oil will be used to remediate a portion of the solvent plume beneath ACP by overcoming the observed electron donor limitation.

The most common approach utilized to date to stimulate reductive dehalogenation has been addition of a carbon source dissolved in groundwater. This approach may prove effective in some applications, but in many cases may have difficulty competing with pump-and-treat remedial systems because the carbon source must be continuously injected. Other approaches involving the emplacement of solid materials that release carbon are promising, but the cost of the solid carbon addition will be high. The separate phase nature of vegetable oil allows for slow dissolution into groundwater, thus making it a slow-release carbon source. Thus, the carbon substrate will only be injected one time, which will significantly reduce overall costs. Vegetable oil is an inexpensive, innocuous, food-or feed-grade carbon source that is not regulated as a contaminant by the USEPA. Because vegetable oil is a non-aqueous phase liquid (NAPL), the potential exists that a

Figure 3.1 Anaerobic Reductive Dehalogenation

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single, low-cost injection could provide sufficient carbon to drive reductive dechlorination for many years.

Vegetable oil will be injected to create the reduction-oxidation (redox) and electron donor conditions necessary to promote the microbial reductive dehalogenation of the chlorinated solvents found beneath ACP. A secondary benefit is partitioning of the dissolved chlorinated solvents into the vegetable oil NAPL. This is beneficial because aqueous-phase chlorinated solvent concentrations will be lowered until steady-state (equilibrium) conditions are reached.

3.2 EVIDENCE FOR BIODEGRADATION AT ANOKA COUNTY PARK

TCE detected at the NIROP facility is assumed to be the parent compound. Degradation daughter products detected in groundwater at the site that are not considered to have been released but are a product of biodegradation include 1,2-DCE and VC.

The presence of 1,2-DCE and low concentrations of VC suggests that reductive dehalogenation is occurring at NIROP and beneath ACP. Based on the low concentrations of 1,2-DCE and VC at these sites it appears that complete bioremediation is limited by a lack of oxidizable organic carbon.

SECTION 4

SYSTEM INSTALLATION AND ADDITIONAL DATA COLLECTION

4.1 FIELD APPLICATION ACTIVITIES

Field activities to be completed for this investigation include initial system installation, baseline sampling, aquifer testing, vegetable oil injection, and groundwater monitoring. Site-specific activities for the initial field event are summarized in Table 4.1, and will include:

- Installation of 1 new background monitoring well in the vicinity of existing monitoring well MS-46S, 3 vegetable oil injection wells, 9 groundwater monitoring wells, and 3 contingency monitoring wells using the hollow stem auger drilling technology (see Section 4.3);
- Background (i.e., pre-injection) sampling of groundwater at the newly installed injection wells, groundwater monitoring wells, contingency monitoring wells, and existing monitoring wells MS-46S, 27S, and MS-47S in accordance with the *Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater* (USEPA, 1998) as specified in Table 4.1 (see Section 4.4);
- Pre-injection aquifer testing (hydraulic conductivity) of the 3 injection wells, 6 groundwater monitoring wells, and one contingency well, if needed (see Section 4.5);
- Plumbing of the vegetable oil injection wells and injection of up to 3,600 gallons of food-grade vegetable oil (see Section 4.6);
- Post-injection aquifer testing (hydraulic conductivity) of the previously-tested 3 injection wells (see Section 4.5); and
- Surveying of the newly installed injection and monitoring wells (see Section 4.3.3.7).

Performance monitoring activities are summarized in Table 4.2, and involve post-injection sampling of groundwater and vegetable oil (if present) at the newly installed injection wells, groundwater monitoring, and contingency monitoring wells and existing monitoring wells MS-46S, 27S, and MS-47S, in accordance with the *Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater* (USEPA, 1998) at 2, 5, 8, and 12 months after vegetable oil injection.

TABLE 4.1
SUMMARY OF PROPOSED INITIAL SITE ACTIVITIES (PRE OIL INJECTION)
NIROP FRIDLEY, MINNESOTA

Location	Well Installation	Slug Test Analysis	Water Level Measurement	Soil Analyses: Total Organic Carbon E415.1	Soil Analyses: Bulk Density ASTM - D2937	Soil Analyses: VOCs SW8260B	Vegetable Oil Analyses [a]: VOCs [b] SW8260B	Groundwater Analyses: VOCs SW8260B	Groundwater Analyses: Methane, Ethane, Ethene	Groundwater Analyses: Nitrate + Nitrite (E300.1)	Groundwater Analyses: Total Organic Carbon (SW9060)	Groundwater Analyses: Well Head Analyses [c]	Groundwater Analyses: Mobile Lab Analyses [d]
ExistingMonitoring Well													
MS-46S		X	X					1	1	1	1	1	1
New Monitoring Wells													
VG-MW-1	X	X	X	1	1	1		1	1	1	1	1	1
VG-MW-2	X		X					1	1	1	1	1	1
VG-MW-3	X	X	X	1	1	1		1	1	1	1	1	1
VG-MW-4	X	X	X	1	1	1		1	1	1	1	1	1
VG-MW-5	X	X	X	1	1	1		1	1	1	1	1	1
VG-MW-6	X		X					1	1	1	1	1	1
VG-MW-7	X		X					1	1	1	1	1	1
VG-MW-8	X	X	X					1	1	1	1	1	1
VG-MW-9	X		X					1	1	1	1	1	1
Existing Contingency Wells													
27S			X					1	1	1	1	1	1
MS-47S			X					1	1	1	1	1	1
New Contingency Wells													
VG-CW-1	X		X					1	1	1	1	1	1
VG-CW-2	X	X	X					1	1	1	1	1	1
VG-CW-3	X		X					1	1	1	1	1	1
Injection Wells													
VG-INJ-1	X	X	X	1	1	1		1	1	1	1	1	1
VG-INJ-2	X	X	X	1	1	1	1	1	1	1	1	1	1
VG-INJ-3	X	X	X	1	1	1		1	1	1	1	1	1
SUBTOTALS	15	10	18	7	7	7	1	18	18	18	18	18	18
QA/QC													
Duplicates						1	1	2	2	2	2		3
MS/MSD						1	1	1					
Trip Blanks						1		5					
Rinseates						1		1					
			TASK TOTAL:	7	7	10	3	27	20	20	20	18	21

[a] Baseline vegetable oil sample will be collected from oil delivered to the site, prior to injection.

[b] VOCs to include aromatic and chlorinated aliphatic hydrocarbons.

[c] Well head analyses include dissolved oxygen, oxidation-reduction potential, pH, temperature, and conductivity.

[d] Mobile lab analyses include chloride, carbon dioxide, alkalinity, sulfate, ammonia, ferrous iron, hydrogen sulfide, and manganese.

E - Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020. (EPA, 1983)

SW - Test Methods for Evaluating Solid Wastes, SW846, Third Edition, (EPA, 1995b)

ASTM - American Society for Testing Materials

TABLE 4.2
SUMMARY OF PROPOSED PERFORMANCE MONITORING ACTIVITIES (Per Sampling Event)
NIROP FRIDLEY, MINNESOTA

Location	Water Level Measurement	Slug Test Analyses	Oil Analyses	Groundwater Analyses					
			VOCs[a] SW8260B	VOCs SW8260B	Methane, Ethane, Ethene	Nitrate + Nitrite (E300.1)	Total Organic Carbon (SW9060)	Well Head Analyses[b]	Mobile Lab Analyses[c]
Existing Monitoring Well									
MS-46S	X			1	1	1		1	1
New Monitoring Wells									
VG-MW-1	X			1	1	1	1	1	1
VG-MW-2	X			1	1	1	1	1	1
VG-MW-3	X			1	1	1	1	1	1
VG-MW-4	X			1	1	1	1	1	1
VG-MW-5	X			1	1	1	1	1	1
VG-MW-6	X			1	1	1	1	1	1
VG-MW-7	X			1	1	1	1	1	1
VG-MW-8	X			1	1	1	1	1	1
VG-MW-9	X			1	1	1	1	1	1
Existing Contingency Wells									
27S	X			1	1	1	1	1	1
MS-47S	X			1	1	1	1	1	1
New Contingency Wells									
VG-CW-1	X			1	1	1	1	1	1
VG-CW-2	X			1	1	1	1	1	1
VG-CW-3	X			1	1	1	1	1	1
Injection Wells									
VG-INJ-1	X	X	1	1	1	1	1	1	1
VG-INJ-2	X	X	1	1	1	1	1	1	1
VG-INJ-3	X	X	1	1	1	1	1	1	1
SUBTOTALS	18	3	3	18	18	18	17	18	18
QA/QC									
Duplicates			1	2	2	2	2		2
MS/MSD			1	2					
Trip Blanks				5					
Rinseates				1					
SUBTOTAL PER EVENT:		3	5	28	20	20	19	18	20
TASK TOTAL:		3	20	112	80	80	76	72	80

[a] VOCs to include aromatic and chlorinated aliphatic hydrocarbons. These samples will be collected only if vegetable oil is present in the injection well.
[b] Well head analyses include dissolved oxygen, oxidation-reduction potential, pH, temperature, and conductivity.
[c] Mobile lab analyses include chloride, carbon dioxide, alkalinity, sulfate, ammonia, ferrous iron, hydrogen sulfide, and manganese.
Note: Hydrogen will be sampled at MS-46S during all performance monitoring events, and during the 5 and 12 month events only in the remainder of the wells.
E - Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020. (EPA, 1983)
SW - Test Methods for Evaluating Solid Wastes, SW846, Third Edition, (EPA, 1995b)

4.2 SITE MANAGEMENT

The following paragraphs outline site management issues pertaining to the field activities to be conducted at ACP. All field activities will be performed by CH2M Hill Constructors, Inc. or their subcontractor. A representative from Parsons ES will be present at the site during system installation, vegetable oil injection, and process monitoring to help CH2M Hill Constructors, Inc. accomplish the field application. Two weeks prior to the commencement of field work related to this program, Paul Estuesta (MPCA On Site Inspector) will be informed of the upcoming field work.

4.2.1 NIROP Support

NIROP will provide the following support during field activities:

- **Assign Accumulation Points.** Any purge fluids and decontamination rinsate/solvents or drill cuttings generated during site work will be properly contained as specified in the Waste Management Plan (CH2M Hill Constructors, Inc, 2000a). The location for the storage of containerized waste at NIROP will be coordinated through UDLP. Waste handling procedures are outlined in Section 4.2.4.

- **Underground Utility Clearances.** Before any field work is conducted, each proposed intrusive sampling location at ACP will be checked for underground utilities. Underground utilities clearances will be obtained through the Gopher State One Call program and clearance will be documented. Any available utility maps will be obtained from Anoka County Park and kept at the site where drilling work will be conducted.

- **Badge and Vehicle Passes.** Personnel badges and vehicle passes will be issued as necessary for field personnel to access equipment staging and decontamination areas at NIROP.

4.2.2 Anoka County Support

- **Access**. Anoka County Park will allow field teams to enter the park and conduct work for the various stages of the project (installation, oil injection, aquifer testing, and monitoring (for the baseline, 2 month, 6 month, and 12 month events).

4.2.3 Contingency Plans

This subsection describes steps that will be taken by CH2M Hill Constructors, Inc. to minimize delays during the investigations. Potential problems that could be encountered during the field effort include:

- Access and coordination difficulties;

- Equipment breakdowns;

- Conflicts with planned sampling locations;
- Abnormal site conditions (e.g., severe weather); and/or
- Monitoring or injection well permit delays.

4.2.3.1 Access and Coordination Contingencies

Anticipated support needs are outlined in Sections 4.2.1 and 4.2.2. In the event that site access difficulties arise, Anoka County Park (ACP) personnel will be contacted to resolve the problem. The site manager and field team leader for CH2M Hill Constructors, Inc. will be responsible for notifying ACP personnel of access or coordination difficulties and working with ACP personnel to rectify any problems that may arise.

4.2.3.2 Equipment Contingencies

In the event of operation problems with field equipment or testing instruments, the following actions will be taken:

- Contact the field team leader;
- Refer to the instrument's instruction book for troubleshooting procedures; and
- Contact the manufacturer and/or supplier.

If necessary, backup instruments will be obtained. However, any such decisions will be made by the site manager for CH2M Hill Constructors, Inc. after consideration of other potential solutions. Equipment will be maintained and extra batteries and other standard replacement parts will be carried in order to avoid downtime due to minor problems.

4.2.3.3 Sampling Location Contingencies

During the field effort, certain chosen sampling locations may be inaccessible due to site conditions. When the conditions can be adjusted the CH2M Hill Constructors, Inc. site manager and/or field team leader will arrange for access to the sampling location. Significant changes in well locations will be approved by MPCA/USEPA before drilling commences.

4.2.3.4 Monitoring and Injection Well Permit Delays

Well construction and underground injection control (UIC) permits (for vegetable oil) will be obtained by CH2M Hill Constructors, Inc. Drilling operations will not begin until these permits are approved. The well and UIC permits will be effective only for the time period indicated by the final signature authority. Reauthorization from all organizations and the technical representative shall be required for any additional time required after expiration of the original permitting period.

4.2.3.5 Project Health and Safety Plan

A site-specific Project Health and Safety Plan Addendum (Appendix A) has been prepared for field activities to be conducted at ACP. The Plan Addendum is designed to augment *Health and Safety Plan Naval Industrial Reserve Ordnance Plant Fridley, Minnesota* (CH2M Hill Constructors, Inc., 2000b) by adding sections pertaining to hazards specific to this pilot-scale study.

4.2.4 Waste Handling

Investigation-derived waste (IDW) will include soil from drilling, groundwater removed from monitoring and injection wells during development or purging, small quantities of solvent contaminated vegetable oil, water and solvents used for decontamination, and some personal protective equipment. CH2M Hill Constructors, Inc. will be responsible for containerizing and arranging for disposal of all IDW generated during the field application. IDW generated during this pilot study will be handled in accordance with waste management requirements presented in *Work Plan Addendum 01, Modification to the Extraction System and Abandonment of Production Wells, Naval Industrial Reserve Ordnance Plant Fridley, Minnesota* (CH2M Hill Constructors, Inc., 2000a). Tim Ruda (UDLP) will be contacted prior to the start of field work related to this program to ascertain existing waste management protocols.

Purge water that is visibly contaminated with vegetable oil will be containerized separately and characterized prior shipped off site to an approved waste disposal facility.

4.3 INITIAL SYSTEM INSTALLATION

The procedures for installing the injection and monitoring wells for the pilot study are discussed in the following subsections

4.3.1 Injection Well and Groundwater Monitoring Well Layout

The proposed well layout consists of 1 new background monitoring well in the vicinity of existing monitoring well MS-46S, 3 injection wells, 9 groundwater monitoring wells, and 3 contingency monitoring wells as shown in Figure 4.1. A cross-sectional view of the proposed wells also is shown in Figure 4.1. These locations were determined from a review of available site data as described in Section 2 and discussions with personnel from CH2M Hill Constructors, Inc., the USEPA, the MPCA, Anoka County Parks, TechLaw, Tetra Tech NUS, the Minnesota Department of Health, and Southern Division, Naval Facilities Engineering Command. The injection well locations were selected to provide distribution of oil in the portion of the plume with the highest TCE concentrations. The 3 injection wells will be located 15 feet apart and centered 20 feet downgradient of existing monitoring well MS-46S. The final well layout may vary significantly from what is shown in Figure 2 as a result of information discovered during the field program. For example, the direction of groundwater flow may be modified when the new extraction wells are placed on-line in Spring 2001, and the well layout may need to be adjusted to accommodate for these changes. The vegetable oil injection pilot testing system will not be installed until after the new extraction wells are brought online and the effects of these wells on the groundwater flow regime is assessed.

The proposed well construction details and screened intervals are listed in Table 4.3. It is important to note that the total depths, screened intervals, and feet of riser listed in Table 4.3 are approximations. Actual depths and length specifications will be determined in the field. The most important completion detail is that the top of the screen for injection wells, groundwater monitoring wells, and contingency monitoring wells will be located 4 feet below the observed groundwater table. The volume of oil to be injected was calculated assuming an even distribution of vegetable oil over a vertical distance of 10 feet and a radius of 5 feet assuming isotropic, homogeneous conditions and an effective porosity of 20 percent. It is anticipated that the radius of influence of the injected vegetable oil will be somewhat greater than the assumed 5 feet (horizontally).

Monitoring wells will be located at distances of 15, 30, 50, 80 and 140 feet along the flowpath downgradient from injection well 2, and additional groundwater monitoring wells are located downgradient from injection wells 1 and 3 as shown in Figure 4.1 Monitoring well locations are intended to monitor the groundwater system immediately downgradient of the injection zone over a period of 12 months. One upgradient well MS-46S is located to monitor background groundwater geochemistry. The three new contingency monitoring wells are to be located approximately 200 feet downgradient from the injection wells. In addition two existing monitoring wells will be utilized as contingency monitoring wells (wells 27S and MS-47S); these wells and the proposed new wells are shown on the potentiometric map in Figure 2.2.

The wells will be installed in stages in order to collect water level elevation data from the first few wells installed to more accurately define the direction of groundwater flow and to better locate the remaining wells hydraulically downgradient.

All well installation and development activities will be performed by a driller licensed in Minnesota.

4.3.2 Drilling and Soil Sampling Procedures

Drilling in unconsolidated soils will be accomplished using the hollow stem auger drilling method to approximate depths specified in Table 4.3. All wells are to be completed with the top of the well screen located approximately 4 feet below the observed groundwater table. A field scientist for CH2M Hill Constructors, Inc. will be responsible for collecting soil samples, maintaining a detailed descriptive log of all subsurface materials recovered during drilling (see Appendix C for an example Geologic Log Form), and properly labeling and storing samples. During borehole advancement, soil samples for visual description will be collected at a frequency sufficient to identify the depths of significant stratigraphic contacts or other soil properties. Soil samples will be collected from drilling cores obtained by hollow stem auger drilling method using a 24-inch long, 2-inch diameter split spoon sampler. Procedures will be modified, if necessary, to ensure good sample recovery.

As each boring is advanced one soil sample will be collected from close vertical proximity to the center of the screened interval to be installed. Aliquots of this soil sample from selected boring locations identified in Table 4.1 will be analyzed for VOCs, bulk density via ASTM Method D2937-94 , and total organic carbon (TOC) via method E415.1 (combustion/oxidation) at an off-site laboratory. Additional aliquots of soil samples

Figure 4.1 Vegetable Oil Injection Pilot Test System Layout

PLEASE DISCARD THIS SHEET AND INSERT THE FIGURE 4.1 FROM YOUR HARD COPY

TABLE 4.3
PROPOSED MONITORING WELL CONSTRUCTION SUMMARY
NIROP FRIDLEY, MINNESOTA

Identification	Well Diameter (inches)	Total Depth (feet bgs)[b/]	Screened Interval (feet bgs)	Feet of Riser (feet)	Feet of Screen (feet)	Interval
New Background Well						
PES-BG-1	3.0	70	30-40	63	10	Shallow Drift
Injection Wells						
PES-INJ-1	3.0	70	30-40	63	10	Shallow Drift
PES-INJ-2	3.0	70	30-40	63	10	Shallow Drift
PES-INJ-3	3.0	70	30-40	63	10	Shallow Drift
Monitoring Wells						
PES-MW-1	3.0	70	30-40	63	10	Shallow Drift
PES-MW-2	3.0	70	30-40	63	10	Shallow Drift
PES-MW-3	3.0	40	30-40	33	10	Shallow Drift
PES-MW-4	3.0	40	30-40	33	10	Shallow Drift
PES-MW-5	3.0	40	30-40	33	10	Shallow Drift
PES-MW-6	3.0	70	30-40	63	10	Shallow Drift
PES-MW-7	3.0	70	30-40	63	10	Shallow Drift
PES-MW-8	3.0	40	30-40	33	10	Shallow Drift
PES-MW-9	3.0	40	30-40	33	10	Shallow Drift
Contingency Wells						
PES-CW-1	2.0	40	30-40	33	10	Shallow Drift
PES-CW-2	2.0	40	30-40	33	10	Shallow Drift
PES-CW-3	2.0	40	30-40	33	10	Shallow Drift

[a/] feet bgs indicates approximate depth in feet below ground surface. All wells will be installed approximately 4 feet below the water table and have 3 feet of riser exposed above ground surface.

will be collected and provided to USGS for microbial analysis, soil isotherm sorption tests and bio-available iron analysis.

After drilling is complete, gamma log geophysical data will be collected from the entire boring at each location.

Detailed geologic logs for each boring and Fence Diagrams or detailed geologic cross sections for the site will be prepared by CH2M Hill or a designated subcontractor for the results report.

Drilling and soil sampling equipment will be decontaminated in accordance with procedures discussed in Section 4.3.3.6.

4.3.3 Injection Well and Groundwater Monitoring Well Installation

This section describes the procedures to be used for installation of the injection and monitoring wells.

4.3.3.1 Pre-Installation Activities

All necessary monitoring and injection well permits will be obtained prior to mobilizing to the field by CH2M Hill Constructors, Inc. In addition, all utility lines will be located, and proposed drilling locations will be cleared prior to any intrusive activities. Responsibilities for these permits and clearances are discussed in Section 4.2.

Water to be used in well installation and equipment cleaning will be obtained by the well drilling Subcontractor from an off-site source. The field scientist will make the final determination as to the suitability of water for these activities.

4.3.3.2 Materials Decontamination

All completion materials will be inspected by the field scientist and determined to be clean and acceptable prior to use. If not factory sealed, casing, screen, end caps, and surface plugs will be cleaned prior to use with a high-pressure, steam/hot-water cleaner using approved water. Materials that cannot be cleaned to the satisfaction of the field scientist will not be used.

4.3.3.3 Screen and Casing

Injection and monitoring wells will be installed through the drill rig drive casing using 2-inch or 3-inch-diameter PVC casing and screen as listed in Table 4.3. Well installation activities will be supervised by CH2M Hill Constructors, Inc. and under the observation of Parsons ES personnel. Injection and monitoring well screens will be constructed of 10 feet of flush-threaded, Schedule 40 PVC with an ID of 2.0-inch or 3.0-inch as listed in Table 4.3. The screens will be factory slotted with 0.020-inch openings. Screens will be installed beneath the water table at the approximate intervals listed on Table 4.3. The casing at each well will be fitted with a PVC bottom cap and a locking top cap.

The field scientist will verify and record the total depth of the injection wells and monitoring wells, the lengths of all casing sections, and the depth to the top of all

completion materials (filter pack, bentonite seal, etc.). All lengths and depths will be measured to the nearest 0.1 foot.

4.3.3.4 Injection and Monitoring Well Completion

One well will be installed in each borehole. A graded sand filter pack will be installed around the screened interval and will extend at least 2 feet above the top of the screen. The sand filter pack will consist of clean 10-20 silica sand. A filter pack seal will be installed above the filter pack using sodium bentonite pellets. The pellet seal will be a minimum of 3 feet thick and will be hydrated in place with potable water if the seal is installed above the water table. The pellet seal will be overlain by a neat cement grout (non-rapid setting cement) that will extend from the top of the pellet seal to approximately 2 feet below the ground surface. The neat cement grout will be overlain by a concrete surface collar that will extend to the ground surface. Each injection and groundwater monitoring well casing will be completed above grade, with a steel protective casing to protect the well. Three two-inch steel ballard posts will be installed around each monitoring well completion to protect the well head.

4.3.3.5 Injection and Monitoring Well Development

The injection wells, groundwater monitoring, and contingency monitoring wells will be developed prior to sampling by CH2M Hill Constructors, Inc. All development activities will be performed by, or overseen by, a Minnesota Licensed driller. Development removes sediment from inside the well casing and flushes fines from the portion of the formation adjacent to the screen. Development will be accomplished using a submersible pump. The pump will be regularly raised and lowered through the screened interval while development is underway so that fines are agitated and removed from the entire screened interval.

Development will continue until a minimum 10 casing volumes of water have been removed from the well and until pH, temperature, specific conductance, DO, and water clarity (turbidity) stabilize. If the water remains turbid, development will continue until the turbidity of the water produced has been stable after the removal of several additional casing volumes.

Development water will be collected in portable poly-tanks. The tanks will be staged in a designated hazardous-waste collection area and disposed of in accordance with the waste management plan (Section 4.2.4). A development record will be maintained for each injection and monitoring well on a form similar to the example in Appendix C. The development record will be completed in the field by the field scientist. Development records will include:

- Injection or groundwater monitoring well number;
- Date and time of development;
- Development method;
- Predevelopment water level and well depth;

- Volume of water produced;
- Description of water produced;
- Post development water level and well depth; and
- Field analytical measurements, including pH, specific conductance, turbidity, temperature, and DO.

4.3.3.6 Equipment Decontamination Procedures

Prior to arriving at the site, and between drilling of each borehole, drill bits, drill pipe, drill casing, instrumented probes, samplers, tools, and other downhole equipment will be decontaminated using a high-pressure, steam/hot water wash. Only potable water will be used for decontamination. All rinsate will be collected in portable tanks or 55-gallon drums and will be staged in a designated location at NIROP. The location for the storage of containerized waste at NIROP will be coordinated through UDLP.

Potable water to be used in equipment cleaning, decontamination, or grouting will be obtained from an offsite water source by the well drilling Subcontractor. Precautions will be taken to minimize any impact to the surrounding area that might result from decontamination operations.

4.3.3.7 Datum Survey

The locations and elevations of the newly installed background, injection and groundwater monitoring wells will be surveyed by a registered surveyor. Horizontal locations will be measured relative to established coordinates. Horizontal coordinates will be measured to the nearest 0.1 foot. The elevation of the ground surface adjacent to the well casing and the measurement datum (top of the casing) will be measured relative to an existing benchmark location. Vertical elevations will be measured with respect to the National Vertical Datum (NAVD) of 1998 to the nearest 0.01 foot. Surveying activities will be coordinated by CH2M Hill Constructors, Inc. The field team may elect to survey the wells in stages, in order to collect water level elevation data from the first few wells installed to more accurately define the direction of groundwater flow and to better locate the remaining wells hydraulically downgradient.

4.4 MEASUREMENT OF BASELINE GEOCHEMICAL CONDITIONS AND CONTAMINANT PROFILES

After installation of the injection, groundwater monitoring, and contingency monitoring wells, CH2M Hill Constructors, Inc., under the observation of Parsons ES, will characterize initial (baseline) site-specific geochemical and contaminant conditions in accordance with the *Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater* (USEPA, 1998). Parsons ES shall evaluate geochemical, metabolic byproduct, and contaminant breakdown product data to evaluate the potential for existing reductive dehalogenation, future enhanced reductive dehalogenation, and any expected changes in the above profile as a result of substrate addition.

TABLE 4.4
ANALYTICAL PROTOCOLS FOR GROUNDWATER SAMPLES
NIROP FRIDLEY, MINNESOTA

MATRIX Analyte	METHOD	FIELD (F) OR ANALYTICAL LABORATORY (L)
WATER		
Redox Potential	Direct-reading meter	F
Dissolved Oxygen	Direct-reading meter	F
pH	Direct-reading meter	F
Specific Conductance	Direct-reading meter	F
Temperature	Direct-reading meter	F
Ferrous Iron	Colorimetric, Hach Method 8146 (or similar)	F
Manganese	Colorimetric, Hach Method 8034 (or similar)	F
Hydrogen Sulfide	Colorimetric, Hach Method 8131 (or similar)	F
Sulfate	Colorimetric, Hach Method 8051 (or similar)	F
Ammonia	Colorimetric, Hach Method 8155 (or similar)	F
Alkalinity (Carbonate [CO_3^{-2}] and Bicarbonate [HCO_3^{-1}])	Titrimetric, Hach Method 8221 (or similar)	F
Chloride	Titrimetric, Hach Kit 8P (or similar)	F
Carbon Dioxide	Titrimetric, CHEMetrics Method 4500 (or similar)	F
Nitrate + Nitrite [as Nitrogen (N)]	E300.1	L
Hydrogen	AM-20-GAX (modified SW8015)	L
Methane, Ethane, Ethene	SW5021	L
Total Organic Carbon	SW9060	L
VOCs[a/]	SW8260B	L
SOIL		
Total Organic Carbon	E415.1	L
VOCs	SW8260B	L
Bulk Density	ASTM D2937	L
VEGETABLE OIL		
VOCs	SW8260B	L

[a/] VOCs = volatile organic compounds.
E - Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020. (EPA, 1983)
SW - Test Methods for Evaluating Solid Wastes, SW846, Third Edition, (EPA, 1995b)
ASTM - American Society for Testing Materials
Analytical laboratory is EnChem for all parameters identified with L; except hydrogen test will be analyzed at Microseeps

Groundwater samples collected from the monitoring locations will be analyzed for oxidation-reduction potential (ORP), DO, pH, specific conductance, temperature, ferrous iron, manganese, hydrogen sulfide, sulfate, alkalinity, carbon dioxide, nitrate + nitrite (as nitrogen [N]), chloride, methane, ethane, ethene, total organic carbon (TOC), and VOCs (Table 4.4).

4.4.1 Groundwater Sampling

Collection of groundwater quality samples will be conducted by qualified scientists and technicians from CH2M Hill Constructors who are trained and experienced in the performance of groundwater sampling, records documentation, and chain-of-custody procedures. One representative from Parsons ES will be onsite during groundwater sample collection and analysis activities. In addition, sampling personnel will have thoroughly reviewed this work plan prior to sample collection and will have a copy of the work plan available onsite for reference. Groundwater samples will be collected from previously installed monitoring wells MS-46S, 27S, and MS-47S and from all newly installed injection, monitoring, and contingency monitoring wells. Groundwater samples collected during this program will be collected using MPCA-approved groundwater sampling techniques (Refer to Appendix G). Well locations that will be sampled during this program are shown on Figure 2.2. Prior to sampling, the injection and monitoring wells will be developed as described in Section 4.3.2.5. In order to maintain a high degree of quality control (QC) during this sampling event, the procedures described in the following sections will be followed.

4.4.2 Preparation for Sampling

All equipment to be used for sampling will be assembled and properly cleaned and calibrated (if required) prior to arriving in the field. In addition, all record-keeping materials will be gathered prior to mobilizing to the field. Sample field log forms are provided in Appendix C.

4.4.2.1 Equipment Cleaning

All portions of sampling and test equipment that will contact the sample matrix will be thoroughly cleaned before each use. This includes the water-level probe and cable, test equipment for onsite use, and other equipment or portions thereof that will contact the samples. Based on the types of sample analyses to be conducted, the following cleaning protocol will be used:

- Wash with potable water and phosphate-free laboratory detergent (Alconox®);
- Rinse with potable water;
- Rinse with isopropyl alcohol;
- Rinse with distilled or deionized water; and
- Air dry the equipment prior to use (to the extent practical).

Any deviations from these procedures will be documented in the field scientist's field notebook and on the groundwater sampling record.

Laboratory-supplied sample containers will be cleaned and sealed by the laboratory. The type of container provided and the method of container decontamination will be documented in the laboratory's permanent record of the sampling event.

4.4.2.2 Equipment Calibration

As required, field analytical equipment will be calibrated according to the manufacturers' specifications prior to field use. This applies to equipment used for onsite measurements of DO, pH, specific conductance, ORP, sulfate, ferrous iron, and other field parameters listed in Table 4.4. All field calibration activities will be documented and all calibration documentation will be included as an appendix in the final results report.

4.4.3 Groundwater Sampling Procedures

Special care will be taken to prevent contamination of the groundwater and extracted samples. The primary way in which sample contamination can occur is through cross-contamination due to insufficient cleaning of equipment between wells. To prevent such contamination, the water-level probe and cable used to determine static water levels and total well depths will be thoroughly cleaned before and after field use and between uses at different sampling locations according to the procedures presented in Section 4.4.2.1. A decontaminated submersible pump and/or bladder pumps will be used for purging and sampling each groundwater sampling location.

A clean pair of new, disposable nitrile or latex gloves will be worn by field personnel each time a different monitoring location is sampled. The following paragraphs present the procedures to be followed for groundwater sample collection. These activities will be performed in the order presented below. Exceptions to this procedure will be noted in the sampler's field notebook and on the groundwater sampling form.

4.4.3.1 Preparation of Location

Prior to starting the sampling procedure, the area around the existing and new monitoring wells will be cleared of foreign materials, such as brush, rocks, and debris. These procedures will prevent sampling equipment from inadvertently contacting debris and potentially becoming contaminated. In addition, the sampling location will be inspected for the integrity of the protective cover, lock, external surface seal, concrete pad, cap, datum reference, and internal surface seal.

4.4.3.2 Water Level and Total Depth Measurements

Prior to removing any water from the sampling well, the static water level will be measured. An electric water-level probe (or oil/water interface probe) will be used to measure the depth to groundwater below the datum to the nearest 0.01 foot. After measuring the static water level, the water-level probe will be slowly lowered to the bottom of the well if the well depth is not known, and the depth will be measured to the nearest 0.01 foot. Based on these measurements, the volume of water to be purged from

the monitoring well will be calculated. Otherwise, previously recorded well depths will be used to calculate purge volumes.

4.4.3.3 Purging Before Sampling

The volume of water contained within the monitoring well casing at the time of sampling will be calculated. Groundwater will be purged from the well until a minimum of 3 well casing volumes are removed and water quality parameters have stabilized. Techniques specified in Appendix G will be used for well purging. Water quality stabilization is defined in Section 4.4.4. Purge waters will be handled in accordance with the procedures outlined in Section 4.2.4.

4.4.3.4 Sample Extraction

Sampling technique specified in Appendix G will be used for sample collection. A submersible and/or bladder pump will be used to extract groundwater samples from the injection and monitoring wells. The sample will be transferred directly into the appropriate sample container. The water will be carefully poured down the inner walls of the sample bottle to minimize aeration of the sample.

Unless other instructions are given by the analytical laboratory, sample containers will be completely filled so that no air space remains in the container. Excess water collected during sampling will be handled according to the procedures outlined in Section 4.2.4.

If a monitoring well is evacuated to a dry state during purging, the monitoring well will be allowed to recharge, and the sample will be collected as soon as sufficient water is present to obtain the necessary sample quantity. Sample compositing or sampling over a lengthy period by accumulating small volumes of water at different times to obtain a sample of sufficient volume will not be allowed.

4.4.4 Onsite Groundwater Parameter Measurement

As indicated on Table 4.4, many of the groundwater chemical parameters will be measured onsite by personnel from CH2M Hill Constructors Inc. under the auspices of Parsons ES personnel. Some of the measurements will be made with direct-reading meters, while others will be made using a Hach or CHEMetrics portable colorimeter or titration kit in accordance with manufacturer-specified procedures. These procedures are described in the following subsections.

Samples will be collected after stable conditions have been reached. Stability will be determined between successive purge volumes equal to one calculated saturated casing volume. Stability is obtained when the temperature, specific conductance, pH, dissolved oxygen, and turbidity fall within the following acceptable ranges: ±0.1 degrees Celsius (°C), ±5 %, ±0.1 pH units, ±2%, and less than 5 ntu respectively.

All glassware or plasticware used in the analyses will have been cleaned prior to sample collection by thoroughly washing with a solution of Alconox and water, and rinsing with distilled water and isopropyl alcohol to prevent interference or cross contamination between measurements. If concentrations of an analyte are greater than the range detectable by the titrimetric method, the analysis will be repeated by diluting the

groundwater sample with distilled water until the analyte concentration falls to a level within the range of the method. All rinsate and sample reagents accumulated during groundwater analysis will be collected in glass containers fitted with screw caps. These waste containers will be clearly labeled as to their contents and carefully stored for later transfer to the approved disposal facility.

4.4.4.1 Dissolved Oxygen Measurements

DO measurements will be made using a meter with a sensor in a flow-through cell or a downhole oxygen sensor. Multiple measurements will be taken before groundwater sample acquisition during well purging, with the final measurement made immediately prior to completion of the well purge. When DO measurements are taken in monitoring wells that have not yet been sampled, the existing monitoring wells will be purged until DO levels stabilize. DO measurements will be recorded on the groundwater sampling record.

4.4.4.2 pH, Temperature, and Specific Conductance

Because the pH, temperature, and specific conductance of a groundwater sample can change significantly within a short time following sample acquisition, these parameters will be measured in the field in a flow-through cell during the purging process as described for DO in Section 4.4.4.1. The measured values will be recorded in the groundwater sampling record.

4.4.4.3 Carbon Dioxide Measurements

Carbon dioxide is a byproduct of biological reactions and can be used to evaluate the bioactivity of the groundwater system. Carbon dioxide concentrations in groundwater will be measured in the field by experienced personnel from CH2M Hill Constructors Inc. under the auspices of Parsons ES personnel via titrimetric analysis using CHEMetrics Method 4500 [0 to 250 mg/L], or equivalent.

4.4.4.4 Alkalinity Measurements

Alkalinity in groundwater helps buffer the groundwater system against acids generated through both aerobic and anaerobic biodegradation processes. Alkalinity of the groundwater sample will be measured in the field by experienced personnel from CH2M Hill Constructors Inc. under the auspices of Parsons ES personnel via titrimetric analysis using USEPA-approved Hach Method 8221 (0 to 5,000 mg/L as calcium carbonate), or equivalent.

4.4.4.5 Nitrate + Nitrite (as Nitrogen) Measurements

Nitrate + nitrite (as N) concentrations are of interest because nitrate can act as an electron acceptor during hydrocarbon biodegradation under anaerobic soil or groundwater conditions. Nitrate is also a potential nitrogen source for hydrocarbon-degrading bacteria biomass formation. Nitrite is an intermediate byproduct in both ammonia nitrification and in nitrate reduction in anaerobic environments.

Nitrate + nitrite (as N) concentrations in groundwater will be measured in the laboratory (Table 4.4). In addition, nitrate and nitrite may be measured in the field by experienced Parsons ES scientists via colorimetric analysis using a Hach DR/700 Portable Colorimeter. Nitrate concentrations in groundwater samples will be analyzed after preparation with Hach Method 8039 (0 to 30.0 mg/L nitrate), or equivalent. Nitrite concentrations in groundwater samples will be analyzed after preparation with USEPA-approved Hach Method 8507 (0 to 0.35 mg/L nitrite), or equivalent.

4.4.4.6 Sulfate and Hydrogen Sulfide Measurements

Sulfate in groundwater is a potential electron acceptor for fuel-hydrocarbon biodegradation in anaerobic environments, and sulfide is produced during sulfate reduction. Sulfate will be measured in the laboratory (Table 4.4), or personnel from CH2M Hill Constructors Inc. under the auspices of Parsons ES personnel may measure sulfate and hydrogen sulfide concentrations via colorimetric analysis with a Hach DR/700 Portable Colorimeter after appropriate sample preparation. USEPA-approved Hach Methods 8051 (0 to 70.0 mg/L sulfate) and 8131 (0.60 mg/L hydrogen sulfide) or equivalent will be used to prepare samples and analyze sulfate and hydrogen sulfide concentrations, respectively.

4.4.4.7 Ferrous Iron Measurements

Iron is an important trace nutrient for bacterial growth, and different states of iron can affect the ORP of the groundwater and act as an electron acceptor for biological metabolism under anaerobic conditions. Ferrous iron concentrations will be measured in the field via colorimetric analysis with a Hach DR/700 Portable Colorimeter after appropriate sample preparation. Hach Method 8146, or equivalent, for ferrous iron (0 to 3.0 mg/L) will be used to prepare and quantitate the samples.

4.4.4.8 Manganese Measurements

Manganese is a potential electron acceptor in anaerobic environments. Manganese concentrations will be quantified in the field using colorimetric analysis with a Hach DR/700 Portable Colorimeter. USEPA-approved Hach Method 8034 (0 to 20.0 mg/L), or equivalent, will be used to prepare the samples for analysis of manganese concentrations.

4.4.4.9 Oxidation/Reduction Potential

The ORP of groundwater is an indicator of the relative tendency of a solution to accept or transfer electrons. Redox reactions in groundwater are usually biologically mediated; therefore, the ORP of a groundwater system depends upon and influences rates of biodegradation. ORPs can be used to provide real-time data on the location of the contaminant plume, especially in areas undergoing anaerobic biodegradation. The ORP of a groundwater sample taken inside the contaminant plume should be somewhat lower than that of a sample taken in an upgradient location.

The ORP of a groundwater sample can change significantly within a short time following sample acquisition and exposure to atmospheric oxygen. Therefore, this parameter will be measured in a flow-through cell as described for DO in Section 4.4.4.1.

4.4.4.10 Chloride Measurements

During biodegradation of chlorinated solvents dissolved in groundwater, chloride is released into the groundwater, resulting in accumulations of biogenic chloride. This results on chloride concentrations in groundwater in the contaminant plume that are elevated relative to background concentrations. In aquifers with relatively low background concentrations of inorganic constituents, the concentration of chloride in the aquifer can be seen to increase as chlorinated solvents are degraded. Chloride concentrations in groundwater will be measured in the field via titrimetric analysis using USEPA-approved Hach Kit 8P or equivalent.

4.4.4.11 Ammonia Measurements

The concentration of ammonia concentration is used as indicator of groundwater chemistry. Often a significant percentage of bioavailable nitrogen in a groundwater system is present as ammonia (NH_3). In addition, as discussed above (Section 6.3.4.5) nitrogen is an important reductive dechlorination byproduct. Nitrogen commonly in the form of ammonia is consumed during reductive dechlorination if the predominant groundwater geochemical conditions are such that denitrification is the dominant dechlorination process present. In the case where denitrification is occurring, nitrogen concentrations in groundwater in the source area will be significantly lower than background concentrations. Ammonia concentrations in groundwater shall be measured in the field using colorimetric analysis with a Hach DR/700 Portable Colorimeter. USEPA-approved Hach Method 8155 or equivalent, will be used to prepare the samples for analysis of ammonia concentrations.

4.4.5 Handling of Samples for Laboratory Analysis

This section describes the handling of samples from the time of sampling until the samples are delivered to the laboratory.

4.4.5.1 Sample Preservation

The laboratory will add any necessary chemical preservatives prior to shipping the sample containers to the field. Samples will be prepared for transportation to the analytical laboratory by placing the samples in a cooler containing ice to maintain a maximum shipping temperature of 4 ºC.

4.4.5.2 Sample Container and Labels

Sample containers and appropriate container lids will be provided by the laboratory (Appendix B). The sample containers will be filled as described in Section 4.4.3.4, and the container lids will be tightly closed. The sample label will be firmly attached to the container side, and the following information will be legibly and indelibly written on the label:

- Facility name;
- Sample station identification;

- ξ Sample type (e.g., groundwater, soil, surface water, sediment);
- ξ Sampling date;
- ξ Sampling time;
- ξ Preservatives added;
- ξ Sample collector's initials; and
- ξ Analyses requested.

4.4.5.3 Sample Shipment

After the samples are sealed and labeled, they will be packaged for transport via an overnight carrier to the laboratory. The packaged samples will be delivered to the laboratory as soon as possible (well within holding limits) after sample collection.

The following packaging and labeling procedures will be followed:

- ξ Package sample so that it will not leak, spill, or vaporize from its container;
- ξ Cushion samples to avoid breakage; and
- ξ Add ice to container to keep samples cool for the duration of the trip to the laboratory.

4.4.5.4 Chain-of-Custody Control

Chain-of-custody documentation for the shipment of samples from the field to the laboratory will be completed.

4.4.5.5 Sampling Records

In order to provide complete documentation of the sampling event, detailed records will be maintained by the field scientist. At a minimum, these records will include the following information:

- ξ Sample location (facility name);
- ξ Sample station identification;
- ξ Date and time of sampling;
- ξ Sampling method;
- ξ Field observations of sample appearance and odor;
- ξ Weather conditions;
- ξ Water level prior to purging (groundwater samples only);

- Total monitoring well/well depth (groundwater samples only);
- Purge volume (groundwater samples only);
- Monitoring well/well condition (groundwater samples only);
- Sampler's identification;
- Field measurements of pH, temperature, DO, ORP, and specific conductance (groundwater samples only); and
- Any other relevant information.

Groundwater sampling information will be recorded on a groundwater sampling form. Sample collection data such as collection location, sample identification number, collection time, and final collection field parameters will be recorded in the project field book.

4.4.5.6 Laboratory Analyses

Laboratory analyses will be performed on all groundwater and soil samples as well as the QA/QC samples described in Section 5. The analytical methods for these sampling events are listed in Table 4.4. Prior to sampling, the laboratory will provide a sufficient number of analyte-specific sample containers for the samples to be collected. All containers, preservatives, and shipping requirements will be consistent with USEPA protocol or those listed in Appendix B of this plan.

Laboratory personnel will specify the necessary QC samples and prepare appropriate QC sample containers. For samples requiring chemical preservation, preservatives will be added to containers by the laboratory. Containers and ice chests with adequate padding will be provided by laboratory personnel. Sampling personnel will fill the sample containers and return the samples to the field laboratory.

4.5 PRE-OIL INJECTION AQUIFER TESTING

Slug tests will be conducted before and after oil injection on the three newly installed injection wells to estimate the hydraulic conductivity of unconsolidated deposits at the site and to estimate the impact of oil injection. In addition, slug tests will be conducted prior to oil injection on seven monitoring wells. Slug testing will be preformed after the baseline geochemical and contaminant sampling.

A slug test is a single-well hydraulic test used to determine the hydraulic conductivity of an aquifer in the immediate vicinity of screened interval of the tested well. Slug tests can be used for both confined and unconfined aquifers that have a transmissivity of less than 7,000 square feet per day (ft^2/day). Slug testing can be performed using either a rising head or a falling head test; at this site, both methods will be used in sequence.

4.5.1 Definitions

- **Hydraulic Conductivity (K).** A quantitative measure of the ability of porous material to transmit water; defined as the volume of water that will flow through a unit cross-sectional area of porous or fractured material per unit time under a unit hydraulic gradient.

- **Transmissivity (T).** A quantitative measure of the ability of an aquifer to transmit water. It is the product of the hydraulic conductivity and the saturated thickness of the water-bearing zone.

- **Slug Test.** Two types of tests are possible: rising head and falling head. A slug test consists of adding a slug of water or a solid cylinder of known volume to the well to be tested or removing a known volume of water or cylinder and measuring the rate of recovery of water level inside the well. The slug of a known volume acts to raise or lower the water level in the well.

- **Rising Head Test.** A test used in an individual well within the saturated zone to estimate the hydraulic conductivity of the surrounding formation adjacent to the screened interval by lowering the water level in the well and measuring the rate of recovery of the water level. The water level may be lowered by pumping, bailing, or removing a submerged slug from the well.

- **Falling Head Test.** A test used in an individual well to estimate the hydraulic conductivity of the surrounding formation adjacent to the screened interval by raising the water level in the well by insertion of a slug or quantity of water, and then measuring the rate of drop in the water level.

4.5.2 Equipment

The following equipment will be used to conduct a slug test:

- Teflon®, PVC, or stainless steel slugs;

- Nylon or polypropylene rope;

- Electric water-level indicator;

- Pressure transducer/sensor;

- Field logbook/forms; and

- Automatic data recording instrument (such as the Hermit Environmental Data Logger®, In-Situ, Inc. Model SE3000, or equivalent).

4.5.3 General Test Methods

Aquifer hydraulic conductivity tests (slug tests) are accomplished by either removal of a slug or quantity of water (rising head) or introduction of a slug (falling head), and then allowing the water level to stabilize while taking water-level measurements at closely

spaced time intervals. Both rising and falling head slug tests will be performed at each well by qualified and experienced CH2M Hill Constructors Inc. personnel.

Because hydraulic testing will be completed on existing wells, it will be assumed that the wells were properly developed and that water levels have stabilized. Slug testing will proceed only after multiple water-level measurements over time show that static water levels are in equilibrium. During the slug test, the water level change should be influenced only by the introduction (or removal) of the slug volume. Other factors, such as inadequate well development or extended pumping, may lead to inaccurate results; in addition, slug tests will not be performed on wells with free product. The field scientist will determine when static equilibrium has been reached in the well. The pressure transducer, slugs, and any other downhole equipment will be decontaminated prior to and immediately after the performance of each slug test using the procedures described in Section 4.4.2.1.

4.5.4 Falling Head Test

The falling head test is the first step in the two-step slug testing procedure. The following steps describe procedures to be followed during performance of the falling head test.

1. Decontaminate all downhole equipment prior to initiating the test.

2. Open the well. Where wells are equipped with water-tight caps, the well should be unsealed at least 24 hours prior to testing to allow the water level to stabilize. The protective casing will remain locked during this time to prevent vandalism.

3. Prepare the aquifer slug test data form (see Appendix C) with entries for:

   - Borehole/well number,
   - Project number,
   - Project name,
   - Aquifer testing team,
   - Climatic data,
   - Top of well casing elevation,
   - Identification of measuring equipment being used,
   - Static water level, and
   - Date.

4. Measure the static water level in the well to the nearest 0.01 foot.

5. Lower the decontaminated pressure transducer into the well and allow the displaced water to return to its static level. This can be determined by periodic

water-level measurements until the static water level in the well is within 0.01 foot of the original static water level.

6. Lower the decontaminated slug into the well to just above the static water level in the well.

7. Turn on the data logger and quickly lower the slug below the water table, being careful not to disturb the pressure transducer. Follow the owner's manual for proper operation of the data logger.

8. Terminate data recording when water level stabilizes in the well. The well will be considered stabilized for termination purposes when it has recovered 80 to 90 percent from the initial displacement.

4.5.5 Rising Head Test

After completion of the falling head test, the rising head test will be performed. The following steps describe the rising head slug test procedure:

1. Measure the water level in the well to the nearest 0.01 foot to ensure that it has returned to the static water level.

2. Initiate data recording and quickly withdraw the slug from the well. Follow the owner's manual for proper operation of the data logger.

3. Terminate data recording when the water level stabilizes in the well, and remove the pressure transducer from the well and decontaminate. The well will be considered stabilized for termination purposes when it has recovered 80 to 90 percent from the initial displacement.

4.5.6 Slug Test Data Analysis

Data obtained during slug testing will be analyzed using the computer code AQTESOLV (Geraghty & Miller, 1994) or similar, and the method of Cooper *et al.* (1967) for confined aquifers or the method of Bouwer and Rice (1976) and Bouwer (1989) for unconfined conditions.

4.6 OIL INJECTION

After the background geochemical and contaminant sampling and slug testing have been completed, an emulsion of 3,600 gallons of food-grade soybean oil/lecithin and 7,200 gallons (total) of native water will be injected into the three new injection wells as specified by below.

The injection system and each injection point will be filled with groundwater to remove entrapped air and to pressure-test the system. Water with similar geochemical characteristics (native) to the site groundwater will be used for the injection to promote the development of a healthy microbial population and avoid adverse changes to site geochemistry. An in-line mixer will be used to emulsify a total of 3,600 gallons of food grade vegetable oil/lecithin mixture with a total of 7,200 gallons of native groundwater

previously extracted from a groundwater extraction well in close proximity to the newly installed injection wells. The oil-lecithin-water emulsion will be pumped into the injection wells until a total of 10,800 gallons of emulsion have been injected into the shallow unconfined aquifer (i.e., 3,600 gallons of oil-lecithin-water emulsion into each well). The in-line mixer will be operated with water for a period after injection is complete to flush that portion of the system. Native groundwater will continue to be injected at ~3.0 gpm until ~60 gallons of native groundwater per well has been injected. This post injection water push will serve to distribute the emulsion further into the aquifer in the immediate vicinity of the injection wells. After the process has been completed, the presence of phase-separated oil emulsion in the injection points and the impact on the groundwater table elevation will be measured with an oil-water interface probe.

The vegetable/lecithin will be purchased from Central Soya Company or other equivalent vendor. The quantity of lecithin added will be determined by Parsons ES personnel.

A temporary mechanical packer will be installed in the injection wells below the screened interval prior to injecting oil+lecithin+water emulsion. The packers will be removed after the emulsion is injected.

4.7 PROCESS MONITORING

In order to monitor system performance over time, personnel from CH2M Hill Constructors Inc. under the auspices of Parsons ES personnel will sample the 3 injection wells, the 9 newly installed groundwater monitoring wells, the 3 newly installed contingency monitoring wells and wells MS-46S, 27S, and MS-47S for the parameters listed in Table 4.4 at 2, 5, 8, and 12 months after oil injection. Based upon the results of the field application, continued monitoring and further injection of vegetable oil will be evaluated. The procedures listed in Section 4.4 will be used for all sampling events.

SECTION 5

QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance/quality control procedures are described in a site-specific QAPP that was developed to address the data needs of this project (Appendix D). The following is a brief discussion of the field QC samples to be collected, the laboratory QC samples required, and the data review procedures that will be performed.

5.1 Field QA/QC Procedures

Field QA/QC procedures will include collection of field duplicates/replicates and rinsate, field, and trip blanks; decontamination of all equipment that contacts the sample medium before and after each use; use of analyte-appropriate containers; and chain-of-custody procedures for sample handling and tracking. All samples to be transferred to the laboratory for analysis will be clearly labeled to indicate sample number, location, matrix (e.g., groundwater), and analyses requested. Samples will be preserved in accordance with the analytical methods to be used, and water sample containers will be packaged in coolers with ice to maintain a temperature of 4 degrees Celsius (θC) or less.

All field sampling activities will be recorded in a bound, sequentially-paginated field notebook in permanent ink. All sample collection entries will include the date, time, sample locations and numbers, notations of field observations, and the sampler's name and signature. Field QC samples will be collected in accordance with the program described below, and as summarized in Table 5.1.

QA/QC sampling will include collection and analysis of duplicate groundwater samples, rinsate blanks, field/trip blanks, and matrix spike samples. Internal laboratory QC procedures will involve the analysis of laboratory control samples (LCSs) and laboratory method blanks (LMBs). QA/QC objectives for each of these samples, blanks, and spikes are described below.

Duplicate groundwater samples will be collected at a frequency of one for every 10 or fewer samples of similar matrix. Each duplicate water sample will be collected concurrently with, and by the same method as, the primary sample. Duplicate water samples will be analyzed for VOCs and geochemical parameters.

One rinsate sample will be collected for every 20 or fewer groundwater samples collected. The rinsate sample will consist of a sample of distilled water poured into or through the sampling device and subsequently transferred into a sample container provided by the laboratory. If dedicated sampling equipment is used, then only one rinsate sample will be collected per sampling device type to document the cleanliness of the dedicated equipment. Rinsate samples will be analyzed for VOCs only.

TABLE 5.1
QA/QC SAMPLING PROGRAM
NIROP FRIDLEY, MINNESOTA

QA/QC Sample Type	Minimum Frequency to be Collected and Analyzed [a/]	Analytes
Duplicates	1 every 10 or fewer samples of similar matrix	VOCs, methane, ethane, ethene, geochemical
Rinseate Blanks	1 per day on re-usable equipment	VOCs
Field Blanks	1 every 20 or fewer groundwater samples	VOCs
Trip Blanks	One per sample shipment containing VOC samples	VOCs
Matrix Spike Samples	5 percent of groundwater and soil samples	VOCs
Laboratory Control Sample	One per method per medium per analytical batch	Laboratory control charts (Method Specific)
Laboratory Method Blanks	One per method per medium per analytical batch	Laboratory control charts (Method Specific)

[a/] Actual frequency of QA/QC samples may be altered by CCI field scientist, but will not be less than minimum QA/QC sampling frequency.

One field blank will be collected for every 20 or fewer groundwater samples to assess the effects of ambient conditions in the field. The field blank will consist of a sample of distilled water poured into a laboratory-supplied sample container at the well while sampling activities are underway. The field blank will be analyzed for VOCs only.

A trip blank will be analyzed to assess the effects of ambient conditions on sampling results during the transportation of samples. The trip blank will be prepared by the laboratory, and will be transported inside each sample shipment containing samples for VOC analysis. Trip blanks will be analyzed for VOCs only.

5.2 Laboratory QC Procedures

Matrix spikes will be prepared in the laboratory and used to establish matrix effects for samples analyzed for VOCs. Sufficient extra sample volume will be submitted to the laboratory to allow matrix spike preparation and analysis. LCSs and LMBs will be prepared internally by the laboratory and will be analyzed each day samples from the sites are analyzed. Samples will be reanalyzed in cases where the LCS or LMB are out of the control limits. Control charts for LCSs and LMBs will be developed by the laboratory and monitored for the analytical methods used (Table 4.4).

5.3 Data Review Procedures

Upon receipt of the data report(s) from the laboratory, 10 percent of the analytical data will be validated by experienced personnel against predetermined criteria to determine whether data quality meets project requirements. The laboratory QC data and the field QC data will be evaluated to objectively ascertain the total level of data quality. Any issues requiring clarification by the laboratory or the samplers will be identified and pursued at this point. Data quality will then be compared to project requirements. If problems are found, qualification of the affected data points will be recommended. After the quality level for each data point is confirmed, data interpretation and evaluation of site contamination will be performed.

The objective of evaluating the quality of the chemical data is to determine if data qualification is necessary. Evaluation will be based upon assessment of the laboratory QC data, the field QC data, and the project DQOs presented in the QAPP. The first step will be to perform an evaluation of the laboratory QC data, a process often termed "data validation." This will provide a rating of the quality of each data point produced by the laboratory. Then the evaluation will compare the quality of the data acquired (determined in the first two steps) to the project's DQOs to determine whether the data are useful. Each step will be completely documented. The overall goal of the data quality evaluation process is to determine whether the data can be used to satisfy project objectives.

SECTION 6

CONTINGENCY PLAN

Refer to the project-specific DQO Notes (Attachment 2 of Appendix D) for specific decision rules, action levels, and criteria for implementing a contingency remedy.

The NIROP partnering team will be consulted to determine if the implementation of a contingency remedy is necessary. If a contingency plan must be implemented, the remedy will likely utilize both the contingency wells installed under this program as well as additional wells installed for the contingency remedy. This remedy will be designed to insure that the discharge of VC to the Mississippi River does not exceed current regulatory guidelines. The final contingency remedy will be designed based upon discussions with the appropriate regulatory personnel. It is anticipated that the selected contingency remedy will involve addition of oxygen to the aquifer to stimulate oxidation of VC. This could be achieved by injecting an oxygen releasing compound (ORC) into selected wells, air sparging, or other oxygenation methods.

SECTION 7

DATA ANALYSIS AND REPORT

Parsons ES will compile, analyze, and interpret field test data in a Field Application Results Report, which will be consistent with the DQOs established for the project (Refer to the project-specific DQO Notes - Attachment 2 of Appendix D). Parsons ES will provide defensible conclusions regarding, but not limited to: the efficiency of electron donor utilization for reductive dehalogenation as compared to metabolic (e.g. methane production) and anabolic (i.e. biomass) processes; contributions or effects of any reagent added to the system (e.g. vegetable oil); extent and uniformity of reagent distribution (e.g. vegetable oil); loss of electron donor and tracer compounds; effective radii of influence; apparent electron donor requirements; observed changes in site-geochemistry; actual/significant changes in contaminant concentrations and mass (considering volatilization, dilution, degradation, and daughter product formation and persistence); reaction kinetics and contact time; feasibility and relative cost-effectiveness of expanded-scale implementation. Based upon the results of the field application, continued monitoring and further injection of vegetable oil will be evaluated.

SECTION 8

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APPENDIX A

PROJECT HEALTH AND SAFETY PLAN ADDENDUM

ADDENDUM TO THE
CH2M HILL HEALTH AND SAFETY PLAN
NAVAL INDUSTRIAL RESERVE ORDNANCE PLANT
FRIDLEY, MINNESOTA

June 2000

February 2001

Prepared by:

Parsons Engineering Science, Inc.
1700 Broadway, Suite 900
Denver, Colorado 80290

REVIEWED AND APPROVED BY:

	Name	Date
Project Manager	Mary C. Stauffer	2/28/01
Program Health & Safety Manager	JA Blakemore for Timothy S. Mustard	02.28.01

1.0 INTRODUCTION

This addendum modifies the existing health and safety plan (HASP) entitled *Health and Safety Plan, Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota* (CH2M Hill Constructors, Inc., 2000) developed for activities related to the modification of the extraction system at the Naval Industrial Reserve Plant (NIROP), Fridley, Minnesota.

This addendum to the CH2M Hill health and safety plan was prepared to address upcoming tasks at Anoka County Park, Fridley, Minnesota. Included or referenced in this addendum are the scope of services, site-specific description and history, project team organization, hazard evaluation of known or suspected chemicals, evaluation of physical hazards, emergency contact information, personal protection levels and equipment, frequency and types of air monitoring equipment, and site control measures. All other applicable portions of the CH2M Hill HASP remain in effect by the CH2M Hill field team leader or health and safety officer.

Site-specific health and safety briefings will be conducted daily prior to the commencement of field activities to communicate the site-specific hazards, activities, and procedures to all field personnel. Documentation of training and briefings, including agenda and signatures of attending personnel, will be maintained onsite by the CH2M Hill field team leader or health and safety officer.

2.0 SCOPE OF SERVICES

Work to be performed includes the installation of vegetable oil injection wells, groundwater monitoring wells, and contingency monitoring wells using a hollow-stem auger drilling rig; aquifer testing; vegetable oil injection into the injection wells; and the collection of soil and groundwater samples by CH2M Hill. Parsons ES will provide field oversight of well drilling and installation, vegetable oil injection, and sampling.

3.0 SITE DESCRIPTION AND HISTORY

The description and history of the site are outlined in the work plan entitled *Work Plan for Field Application to Enhance In-Situ Bioremediation of Chlorinated Solvents via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota* (Parsons ES, 2001).

4.0 PROJECT TEAM ORGANIZATION

The site health and safety office for CH2M Hill will be responsible for the implementation of the health and safety activities at the site. Dan Griffiths, or another qualified Parsons ES employee, will provide Parsons ES oversight on this project and will function as the Parsons ES site health and safety officer.

5.0 HAZARD EVALUATION

5.1 Chemical Hazards

Potential chemical hazards are addressed in the program HASP. Site-specific hazards are identified below.

Trichloroethene (TCE) is the primary contaminant of concern at Anoka County Park. Other chlorinated solvents such as 1,1-dichloroethene (1,1-DCE), vinyl chloride, and tetrachloroethene (PCE) are also of concern.

Health hazard qualities for each of the compounds listed above are presented in Table 1 of this addendum. If additional compounds are discovered during the course of field activities, this health and safety plan addendum shall be amended and pertinent information about the compounds will be communicated to all field personnel. Material Safety Data Sheets (MSDSs) for calibration gases for air monitoring instruments will be filed and maintained onsite by CH2M Hill for reference to safety hazards and storage criteria.

5.2 Physical Hazards

Potential physical hazards at this site include risks associated with the hollow-stem auger drilling rig; soil and groundwater sampling activities; motor vehicles; slip, trip, and fall hazards; noise; and cold exposure. Safe work practices related to the site physical hazards are contained in the CH2M Hill HASP and below.

5.2.1 Drilling Rigs

Working with large motor vehicles could be a major hazard at these sites. Injuries can result from equipment dislodging and striking unsuspecting personnel, and impacts from flying objects or overturning of vehicles. Vehicles and heavy equipment design and operation will be in accordance with 29 CFR, Subpart O, 1926.600 through 1926.602. In particular, the following precautions will be used to help prevent injuries and accidents:

- Because the drilling activities will occur in a public park, the working area around the drill rig will be barricaded with caution tape, or other suitable barriers, to restrict access to the working area.
- Daily vehicle inspections will be conducted and documented.
- Do not back up large motor vehicles unless the vehicle has backup warning lights and a reverse signal alarm audible above the surrounding noise level, or an observer signals it is safe to do so.
- Motor vehicle cabs will be kept free of all nonessential items and all loose items will be secured.
- Drilling rig masts will be lowered to the ground and parking brakes will be set before shutting off the vehicle.
- Drilling rig brakes, cables, kill switches, hydraulic lines, light signals, fire extinguishers, fluid levels, steering, tires, horn, and other safety devices will be inspected daily.
- All personnel working at and around the drilling rig must be informed of the locations of the kill switches.
- Only qualified operators will be allowed to operate heavy equipment.

TABLE 1 HEALTH HAZARD QUALITIES OF HAZARDOUS SUBSTANCES OF CONCERN

Compound	PEL [a/] (ppm)	TLV [b/] (ppm)	IDLH [c/] (ppm)	Odor Threshold[d/] (ppm)	Ionization Potential[e/] (eV)	Physical Description/Health Effects/Symptoms
1,1-Dichloroethane (DCA)	100	100	3,000	120	11.06	Colorless, oily liquid with chloroform-like odor and hot saccharine taste. Irritates skin. Causes CNS depression and kidney, lung, and liver damage. Experimental teratogen and questionable carcinogen.
1,1-Dichloroethene (DCE) (Vinylidene Chloride)	1	5	NA [f/]	NA	10.00	Colorless liquid or gas (>89°F) with a mild, sweet, chloroform-like odor. Irritates eyes, skin, and throat. Causes dizziness, headaches, nausea, shortness of breath, liver and kidney dysfunctions, and lung inflammation. Mutagen and carcinogen.
1,2-Dichloroethene (DCE) (cis- and trans-isomers)	200	200	1,000	0.085-500	9.65	Colorless liquid (usually a mixture of cis- and trans- isomers), with a slightly acrid, chloroform-like odor. Irritates eyes and respiratory system. CNS depressant. Cis- isomer is a mutagen.
4-Methyl 2-pentanone [Methyl Isobutyl Ketone (MIBK), Hexone]	50	50	500	0.28-8	9.30	Colorless liquid with a fruity, ethereal odor. Irritates eyes, skin, and mucous membranes. Causes dermatitis, headaches, narcosis, and coma. In animals, causes liver and kidney damage. Experimental teratogen.
Tetrachloroethene (PCE) (Perchlorethylene)	25 [g/]	25	150	5-50	9.32	Colorless liquid with a mild chloroform odor. Eye, nose, skin and throat irritant. Causes nausea, flushed face and neck, vertigo, dizziness, headaches, hallucinations, incoordination, drowsiness, coma, pulmonary changes, and skin redness. Cumulative liver, kidney, and CNS damage. In animals, causes liver tumors. Mutagen, experimental teratogen, and carcinogen.
Toluene	100	50 (skin) [h/]	500	0.2-40 [i/]	8.82	Colorless liquid with sweet, pungent, benzene-like odor. Irritates eyes and nose. Causes fatigue, weakness, dizziness, headaches, hallucinations or distorted perceptions, confusion, euphoria, dilated pupils, nervousness, tearing, muscle fatigue, insomnia, skin tingling, dermatitis, bone marrow changes, and liver and kidney damage. Mutagen and experimental teratogen.
1,1,1-Trichloroethane (TCA) (Methyl Chloroform)	350	350	700	20-500	11.00	Colorless liquid with a mild chloroform-like odor. Irritates eyes and skin. Causes headaches, exhaustion, CNS depression, poor equilibrium, dermatitis, liver damage, cardiac arrhythmia, hallucinations or distorted perceptions, motor activity changes, aggression, diarrhea, and nausea or vomiting. Mutagen, experimental teratogen, and questionable carcinogen.

TABLE 1 HEALTH HAZARD QUALITIES OF HAZARDOUS SUBSTANCES OF CONCERN

Compound	PEL [a/] (ppm)	TLV [b/] (ppm)	IDLH [c/] (ppm)	Odor Threshold[d/] (ppm)	Ionization Potential[e/] (eV)	Physical Description/Health Effects/Symptoms
Trichloroethene (TCE)	50	50	1,000	21.4-400	9.45	Clear, colorless or blue liquid with chloroform-like odor. Irritates skin and eyes. Causes fatigue, giddiness, headaches, vertigo, visual disturbances, tremors, nausea, vomiting, drowsiness, dermatitis, skin tingling, cardiac arrhythmia, and liver injury. In animals, causes liver and kidney cancer. Mutagen, experimental teratogen, and carcinogen.
Vinyl Chloride	1 (29 CFR 1910.1017) [j/]	5	NA	260	9.99	Colorless gas (liquid<7°F) with a pleasant odor at high concentrations. Severe irritant to skin, eyes, and mucous membranes. Causes weakness, abdominal pain, gastrointestinal bleeding, enlarged liver, pallor or blue skin on the extremities, liver cancer, and frostbite (liquid). Also attacks lymphatic system. Mutagen, experimental teratogen, and carcinogen.

a/ PEL = Permissible Exposure Limit. OSHA-enforced average air concentration to which a worker may be exposed for an 8-hour workday without harm. Expressed as parts per million (ppm) unless noted otherwise. PELs are published in the *NIOSH Pocket Guide to Chemical Hazards*, 1997. Some states (such as California) may have more restrictive PELs. Check state regulations.

b/ TLV = Threshold Limit Value - Time-Weighted Average. Average air concentration (same definition as PEL, above) recommended by the American Conference of Governmental Industrial Hygienists (ACGIH), *TVLs® and BEIs®, 1999*.

c/ IDLH = Immediately Dangerous to Life or Health. Air concentration at which an unprotected worker can escape without debilitating injury or health effects. Expressed as ppm unless noted otherwise. IDLH values are published in the *NIOSH Pocket Guide to Chemical Hazards*, 1997.

d/ When a range is given, use the highest concentration.

e/ Ionization Potential, measured in electron volts (eV), used to determine if field air monitoring equipment can detect substance. Values are published in the *NIOSH Pocket Guide to Chemical Hazards*, June 1997.

f/ NA = Not available.

g/ NIOSH recommends reducing exposure to the lowest feasible concentration, and limiting the number of workers exposed.

h/ (skin) = Refers to the potential contribution to the overall exposure by the cutaneous route.

i/ Olfactory fatigue has been reported for the compound and odor may not serve as an adequate warning property.

j/ Refer to expanded rules for this compound.

- When heavy equipment is left unattended, loads must be lowered, controls neutralized, power shut off, and brakes set. Wheels must be chocked if a vehicle is parked on an incline.

When working near a drill rig, personnel shall be aware of snag hazards from rotating tools and pinch and crush hazards from suspended tools. No loose, dangling clothing will be allowed. Personnel will also be aware of falling object hazards and wear hard hats at all times. Personnel will be aware of slip, trip, and fall hazards from drilling equipment, tools, and well construction materials that may be lying on the ground in the vicinity of the drill rig. To reduce the threat of slip, trip, and fall hazards, the area immediately around the drill rig will be kept clear of equipment and supplies. Use of a downhole hammer will require the use of an air compressor. The compressor air hoses will be checked daily prior to startup for cracks or other defects that could result in injuries.

5.2.2 Noise-Induced Hearing Loss

Noise levels in the area of the drilling rig will be presumed in exceedance of the OSHA time-weighted average (TWA) limit of 85 decibels (dB), and hearing protection will be required. Foam earplugs will generally provide adequate protection. Ear muffs or disposable foam earplugs will be made available to, and used by, all personnel in the vicinity of the drilling rig or other sources of high intensity noise.

6.0 EMERGENCY RESPONSE PLAN

6.1 Emergency Information

The names and telephone numbers for medical and emergency services in the event of any situation or unplanned occurrence requiring assistance are located in the CH2M Hill HASP. For emergency situations, telephone or radio contact should be made with the site point of contact or site emergency personnel who will then contact the appropriate response team. Parsons ES contacts are listed below. A list of emergency contacts must be posted at the site.

Parsons ES Contacts	Telephone Number
Mary Stauffer Project Manager	(303) 831-8100 or (303) 764-8717 (W) (303) 804-0609 (H)
Dr. Frank L. Mitchell Medical Director, Qualisys	(800) 874-4676 (Phone) (888) 926-1500 (Fax)
Timothy Mustard, C.I.H. Program Health and Safety Manager (Denver)	(303) 831-8100 or (303) 764-8810 (W) (303) 450-9778 (H)
Ed Grunwald, C.I.H. Corporate Health and Safety Manager (Atlanta)	(678) 969-2394 (W) (404) 299-9970 (H)

Judy Blakemore Asst. Program Health and Safety Manager (Denver)	(303) 831-8100 or (303) 764-8861 (W) (303) 828-4028 (H) (303) 817-9743 (M)

7.0 LEVELS OF PROTECTION AND PERSONAL PROTECTIVE EQUIPMENT REQUIRED FOR SITE ACTIVITIES

The personal protection level prescribed for field activities at NIROP is OSHA Level D. Currently, according to the CH2M Hill HASP, Levels B and C are not approved for activities related to this scope of work. However, Parsons protocol for ppe evaluation and upgrade is as follows.

While there is a Dräger® tube for vinyl chloride, there is no Dräger® tube for 1,1-DCE. Therefore the following will occur. If sustained air monitoring readings in the worker breathing zone indicate vapor concentrations greater than or equal to 1 part per million (ppm) above background for 30 seconds or longer, the field crew will be forced to evacuate and ventilate the area until readings are less than 1 ppm in the worker breathing zone. If ventilation is inadequate, air samples will be taken to confirm or deny the existence of the contaminants of concern and/or the crew will upgrade to Level B respiratory protection. These air samples will be sent to a lab to be analyzed by Environmental Protection Agency (EPA) Compendium Method TO-14 or the equivalent. Method TO-14 will also analyze for the other volatile contaminants of concern at the site as listed in Table 1 of this addendum.

If vinyl chloride and/or 1,1-DCE are found to exist in the worker-breathing zone at concentrations above 1 ppm above background, additional work must be performed in OSHA Level B PPE due to the inadequate warning properties of the compounds. If other volatile compounds listed in Table 1 are present as indicated by the TO-14 analytical results, the following will be used to check for the additional compounds.

If sustained air monitoring readings in the worker breathing zone persist at or above 25 ppm, Dräger® tubes or the equivalent must be used to confirm or deny the presence of PCE. Due to the inadequate warning properties of PCE, Level B protection must be used if concentrations of PCE exceed 25 ppm above background in the worker-breathing zone.

If PCE is not present, continue to monitor the air in the worker-breathing zone. If concentrations in the worker-breathing zone persist above 25 ppm above background as indicated by the PID, periodic use of the PCE Dräger® tubes must be used to confirm the absence of PCE.

If the PID indicates concentrations at or above 50 ppm above background in the worker-breathing zone, the screening process must be repeated with trichloroethene Dräger® tubes to confirm or deny the presence of trichloroethene. There is not currently a Dräger® tube for 4-methyl-2-pentanone (PEL = 50 ppm). However, EPA Method TO-14, described above will report the compound.

8.0 FREQUENCY AND TYPES OF AIR MONITORING

Personal exposure monitoring in the worker-breathing zone will be conducted using a PID equipped with an 11.7 electron volt (eV) lamp, based on the ionization potential of

the contaminants of concern. The CH2M Hill HASP specifies a 10.6 eV or equivalent lamp, but this will not be sufficient to detect 1,1-DCA or 1,1,1-trichloroethane.

Dräger® tubes will be used when necessary as described above.

APPENDIX B

ANALYTICAL METHODS, DATA USE AND PACKAGING REQUIREMENTS FOR SOIL AND GROUNDWATER SAMPLES

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ANALYTICAL METHODS, DATA USE, AND PACKAGING REQUIREMENTS FOR SOIL AND GROUNDWATER SAMPLES

Matrix	Analysis	Method/Reference	Comments	Data Use	Recommended Frequency of Analysis	Sample Volume, Sample Container, Sample Preservation	Field or Fixed-Base Laboratory
Soil	Total Organic Carbon	SW9060M modified for soil samples	Procedure must be accurate over the range of 0.1 to 5 percent TOC	Measurement of existing organic carbon in subsurface soils. The rate of migration of petroleum contaminants in groundwater is dependant upon the amount of TOC in the aquifer matrix.	During well installation	Collect 100 grams of soil in a glass container with a Teflon lined cap. Cool to 4°C	Fixed-Base Laboratory
Water	Ferrous (Fe^{+2})	Colorimetric A3500-Fe D	Field only	May indicate an anaerobic degradation process due to depletion of oxygen, nitrate, and manganese.	Each sampling round	Collect 100 ml of water in a glass container; acidify with hydrochloric acid per method	Field
Water	Ferrous (Fe^{+2})	Colorimetric HACH Method # 8146	Alternate method; field only	Same as above.	Each sampling round	Collect 100 ml of water in a glass container	Field
Water	Manganese	Colorimetric HACH Method # 8034	Field only		Each sampling round	Collect 100 mL of water in a glass container	Field
Water	Chloride	Mercuric nitrate titration A4500-Cl^- C	Ion chromatography (IC) method E300 or method SW9050 may also be used	General water quality parameter used as a marker to verify that site samples are obtained from the same groundwater system.	Each sampling round	Collect 250 mL of water in a glass container	Field
Water	Chloride	HACH Chloride test kit model 8-P	Silver nitrate titration	Same as above.	Each sampling round	Collect 100mL of water in a glass container	Field
Water	Oxygen	Dissolved oxygen meter	Refer to method A4500 for a comparable laboratory procedure	The oxygen concentration is a data input to the Bioplume model; concentrations less than 1 mg/L generally indicate an anaerobic pathway.	Each sampling round	Collect 300 mL of water in biochemical oxygen demand bottles; analyze immediately; , or measure continuously through a flow through cell.	Field

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Matrix	Analysis	Method/Reference	Comments	Data Use	Recommended Frequency of Analysis	Sample Volume, Sample Container, Sample Preservation	Field or Fixed-Base Laboratory
Water	Conductivity	E120.1/SW9050; direct reading meter	Protocols/Handbook methods	General water quality parameter used as a marker to verify that site samples are obtained from the same groundwater system.	Each sampling round	Collect 100–250 mL of water in a glass or plastic container, or measure continuously through a flow through cell.	Field
Water	Alkalinity	HACH Alkalinity test kit model AL AP MG-L, method 8221	Phenolphthalein method	General water quality parameter used (1) as a marker to verify that all site samples are obtained from the same groundwater system and (2) to measure the buffering capacity of groundwater.	Each sampling round	Collect 100mL of water in glass container	Field
Water	Alkalinity	A2320; titrimetric; E310.2 or E150.1; colorimetric	Handbook method	Same as above.	Each sampling round	Collect 250 mL of water in a glass or plastic container; analyze within 6 hours.	Field
Water	Nitrate (NO_3^{-1}) and Nitrite (NO_2^{-1})	IC method E300 or method SW9056; colorimetric, method E353.2	Method E300 is a Handbook method; method SW9056 is an equivalent procedure	Substrate for microbial respiration if oxygen is depleted.	Each sampling round	Collect up to 40 mL of water in a glass or plastic container; cool to 4°C; analyze within 48 hours	Fixed-Base Laboratory
Water	Nitrate (NO_3^{-1})	HACH method # 8039 for high range method # 8192 for low range	Colorimetric	Same as above.	Each sampling round	Collect 100mL of water in a glass container.	Field
Water	Nitrite (NO_2^{-1})	HACH method #8040	Colorimetric	Substrate for microbial respiration if oxygen is depleted.	Each sampling round	Collect 100mL of water in a glass container	Field
Water	Sulfate (SO_4^{-2})	IC method E300 or method SW9056	Method E300 is a Handbook method; method SW9056 is an equivalent procedure	Substrate for anaerobic microbial respiration.	Each sampling round	Collect up to 40 mL of water in a glass or plastic container; cool to 4°C	Fixed-Base Laboratory

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Matrix	Analysis	Method/Reference	Comments	Data Use	Recommended Frequency of Analysis	Sample Volume, Sample Container, Sample Preservation	Field or Fixed-Base Laboratory
Water	Sulfate (SO_4^{-2})	HACH method # 8051	Colorimetric	Same as above.	Each sampling round	Collect up to 40 mL of water in a glass or plastic container; cool to 4°C	Field
Water	Dissolved sulfide (S^{-2})	HACH method # 8131	Colorimetric	Product of sulfate-based anaerobic microbial respiration; analyze in conjunction with sulfate analysis.	Each sampling round	Collect 100 mL of water in a glass container; analyze immediately	Field
Water	Carbon dioxide	HACH test kit model CA-23, method 8223 or CHEMetrics Method R-1910	Titrimetric; alternate method	The presence of free carbon dioxide dissolved in groundwater is unlikely because of the carbonate buffering system of water, but if detected, the carbon dioxide concentrations should be compared with background to determine whether they are elevated; elevated concentrations of carbon dioxide could indicate biodegradation of BTEX.	Each sampling round	Collect 100 mL of water in a glass container	Field
Water	Methane, Ehane, and Ethene	Kampbell *et al.* 1989 or SW3810 modified.	Method published and used by the U.S. Environmental Protection Agency (EPA) Nation Risk Management Research Laboratory	The presence of methane suggests hydrocarbon degradation via an anaerobic pathway utilizing carbon dioxide (carbonate) as the electron acceptor (methanogenesis).	Each sampling round	Collect water samples in 50 ml glass serum bottles with butyl gray/Teflon-lined caps; add H_2SO_4 to pH 2; cool to 4°C	Fixed-Base Laboratory

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ANALYTICAL METHODS, DATA USE, AND PACKAGING REQUIREMENTS FOR GROUNDWATER SAMPLES

Matrix	Analysis	Method/Reference	Comments	Data Use	Recommended Frequency of Analysis	Sample Volume, Sample Container, Sample Preservation	Field or Fixed-Base Laboratory
Water	VOCs (including aromatic and chlorinated aliphatic hydrocarbons)	SW8260B	Handbook method; analysis may be extended to higher molecular weight alkyl benzenes	Method of analysis for chlorinated solvents and byproducts, which are the primary target analytes for monitoring natural attenuation; method can be extended to higher molecular weight alkyl benzenes; trimethylben-zenes are used to monitor plume dilution if degradation is primarily anaerobic.	Each sampling round	Collect water samples in 2-40 mL VOA vials; cool to 4°C; add hydrochloric acid to pH 2	Fixed-Base Laboratory
Vegetable Oil	VOCs (including aromatic and chlorinated aliphatic hydrocarbons)	SW8260B		To document baseline concentrations of VOCs in the vegetable oil.	Once, Sample will be collected upon delivery of vegetable oil to the site and prior to injection.	Collect water samples in 2-40 mL VOA vials; cool to 4°C; add hydrochloric acid to pH 2	Fixed-Base Laboratory
Water	Total organic Carbon (TOC)	SW9060		Used to classify the plume and to measure the dissolution and spread of the vegetable oil.	Each sampling round	Collect 100 mL of water in an amber glass container with Teflon-lined cap; preserve with sulfuric acid to pH less than 2; cool to 4°C	Fixed-Base Laboratory
Water	pH	E150.1/SW9040; direct reading meter	Protocols/Handbook methods	Aerobic and anaerobic processes are pH-sensitive.	Each sampling round	Collect 100-250 mL of water in a glass or plastic container; analyze immediately, or measure continuously through a flow through cell.	Field

APPENDIX B
ANALYTICAL METHODS, DATA USE, AND PACKAGING REQUIREMENTS FOR GROUNDWATER SAMPLES

Matrix	Analysis	Method/Reference	Comments	Data Use	Recommended Frequency of Analysis	Sample Volume, Sample Container, Sample Preservation	Field or Fixed-Base Laboratory
Water	Temperature	E170.1	Field only	Well development.	Each sampling round	Collect 100–250 mL of water in a glass or plastic container; analyze immediately, or measure continuously through a flow through cell.	Field
Water	Redox potential	A2580 B	Measurements are made with electrodes; results are displayed on a meter; samples should be protected from exposure to atmospheric oxygen	The redox potential of groundwater influences and is influenced by the nature of the biologically mediated degradation of contaminants; the redox potential of groundwater may range from more than 200 mV to less than -400 mV	Each sampling round	Collect 100–250 mL of water in a glass container; filling container from bottom; analyze immediately	Field

NOTES:

1. "HACH" refers to the HACH Company catalog, 1990.
2. "A" refers to *Standard Methods for the Examination of Water and Wastewater*, 18th edition, 1992.
3. "E" refers to *Methods for Chemical Analysis of Water and Wastes*, U.S. Environmental Protection Agency, March 1979.
4. "Protocols" refers to the AFCEE *Environmental Chemistry Function Installation Restoration Program Analytical Protocols*, 11 June 1992.
5. "Handbook" refers to the AFCEE *Handbook to Support the Installation Restoration Program (IRP) Remedial Investigations and Feasibility Studies (RI/FS)*, September 1993.
6. "SW" refers to the *Test Methods for Evaluating Solid Waste, Physical, and Chemical Methods*, SW-846, U.S. Environmental Protection Agency, 3rd edition, 1986.
7. "ASTM" refers to the *American Society for Testing and Materials*, current edition.
8. "RSKSOP" refers to *Robert S. Kerr (Environmental Protection Agency Laboratory) Standard Operating Procedure*.
9. *International Journal of Environmental Analytical Chemistry*, Volume 36, pp. 249-257, "Dissolved Oxygen and Methane in Water by a Gas Chromatography Headspace Equilibration Technique," by D. H. Kampbell, J. T. Wilson, and S. A. Vandegrift.

APPENDIX C

FIELD FORMS

GEOLOGIC LOG

GEOLOGIC LOG	DATE STARTED: DATE COMPLETED:	LOGGER:	HOLE NO.:
1. COMPANY NAME: PARSONS ENGINEERING SCIENCE, INC.		2. DRILLING SUBCONTRACTOR:	
3. PROJECT:		4. DRILLER/DRILL RIG TYPE:	

USCS CODE	DEPTH	DESCRIPTION OF MATERIALS	ANALYTICAL SAMPLE NO.	PID (ppm)	WELL COMPLETION	COMMENTS
	1					
	2					
	3					
	4					
	5					
	6					
	7					
	8					
	9					
	10					
	11					
	12					
	13					
	14					
	15					

GEOLOGIC LOG

GEOLOGIC LOG	DATE STARTED: DATE COMPLETED:	LOGGER:	HOLE NO.:
1. COMPANY NAME: PARSONS ENGINEERING SCIENCE, INC.		2. DRILLING SUBCONTRACTOR:	
3. PROJECT:		4. DRILLER/DRILL RIG TYPE:	

USCS CODE	DEPTH	DESCRIPTION OF MATERIALS	ANALYTICAL SAMPLE NO.	PID (ppm)	WELL COMPLETION	COMMENTS
	16					
	17					
	18					
	19					
	20					
	21					
	22					
	23					
	24					
	25					
	26					
	27					
	28					
	29					
	30					

GEOLOGIC LOG

GEOLOGIC LOG	DATE STARTED: DATE COMPLETED:	LOGGER:	HOLE NO.:
1. COMPANY NAME: PARSONS ENGINEERING SCIENCE, INC.		2. DRILLING SUBCONTRACTOR:	
3. PROJECT:		4. DRILLER/DRILL RIG TYPE:	

USCS CODE	DEPTH	DESCRIPTION OF MATERIALS	ANALYTICAL SAMPLE NO.	PID (ppm)	WELL COMPLETION	COMMENTS
	31					
	32					
	33					
	34					
	35					
	36					
	37					
	38					
	39					
	40					
	41					
	42					
	43					
	44					
	45					

GEOLOGIC LOG

GEOLOGIC LOG	DATE STARTED: DATE COMPLETED:	LOGGER:	HOLE NO.:
1. COMPANY NAME: PARSONS ENGINEERING SCIENCE, INC.		2. DRILLING SUBCONTRACTOR:	
3. PROJECT:		4. DRILLER/DRILL RIG TYPE:	

USCS CODE	DEPTH	DESCRIPTION OF MATERIALS	ANALYTICAL SAMPLE NO.	PID (ppm)	WELL COMPLETION	COMMENTS
	46					
	47					
	48					
	49					
	50					
	51					
	52					
	53					
	54					
	55					
	56					
	57					
	58					
	59					
	60					

GEOLOGIC LOG	DATE STARTED: DATE COMPLETED:	LOGGER:	HOLE NO.:
1. COMPANY NAME: PARSONS ENGINEERING SCIENCE, INC.		2. DRILLING SUBCONTRACTOR:	
3. PROJECT:		4. DRILLER/DRILL RIG TYPE:	

USCS CODE	DEPTH	DESCRIPTION OF MATERIALS	ANALYTICAL SAMPLE NO.	PID (ppm)	WELL COMPLETION	COMMENTS
	61					
	62					
	63					
	64					
	65					
	66					
	67					
	68					
	69					
	70					
	71					
	72					
	73					
	74					
	75					

WELL DEVELOPMENT REPORT

Parsons ES		WELL #:

PROJECT : _____

LOCATION: _____

DATE: _____

PROJECT NO. : _____

DRILLING METHOD (s): _____

PUMP METHOD (s): _____

SURGE METHOD (s): _____

INSTALLATION DATE: _____

INSPECTOR: _____

CONTRACTOR: _____

CREW: _____

START DEVELOPMENT DATE: _____

END DEVELOPMENT DATE: _____

WATER DEPTH (TOC): _____ ft

WELL DIA. (ID CASING): _____ ft

BORING DIAMETER: _____ ft

INSTALLED POW DEPTH(TOC): _____ ft

MEASURED POW DEPTH(TOC): _____ ft

SILT THICKNESS: _____ ft

POW AFTER DEVELOPMENT: _____ ft

DIAMETER FACTORS (GAL/FT):

DIAMETER (IN):	2	3	4	5	6	7	8	9	10	11	12
GALLONS/ FT:	0.163	0.367	0.654	1.02	1.47	2.00	2.61	3.30	4.08	4.93	5.87

CASING VOLUME INSIDE WELL = WATER COLUMN LENGTH X WELL DIAMETER FACTOR

_____ GAL. (A)

MINIMUM VOLUME TO BE REMOVED = 10 X A

_____ GALS.

ACTIVITY	START TIME	END TIME	ELAPSED TIME	GALLONS REMOVED	pH	CONDUCTIVITY	TEMP	COLOR	OTHER
TOTALS/FINAL									

COMMENTS:

S:\es\shared\griffiths\forms\well_develop_2.xls

Slug Test Report

Parsons Engineering Science | **WELL #:**

PROJECT: _____ INSPECTOR: _____
LOCATION: _____ TEST DATE: _____

DRILLING METHOD(S): _____ INSTALLATION DATE: _____
_____ DEVELOPMENT DATE: _____
(data from well completion and development reports)

WELL INFORMATION

BORING DIAMETER: _____ SLOT SIZE: _____
SCREEN DIAMETER: _____ SAND SIZE: _____
DEPTH TOP OF SCREEN: _____ PRODUCT PRESENT (Y/N?) _____
DEPTH BOTTOM OF SCREEN: _____ STATIC WATER LEVEL: _____
DEPTH POW (INSTALLED): _____ STATIC PRODUCT LEVEL: _____
DEPTH POW (MEASURED): _____
(all depths measured from TOC, or taken from installation detail)

TEST EQUIPMENT

DATA RECORDING METHOD: _____ TRANSDUCER RATING: _____
INSTRUMENT BRAND/MODEL: SLUG/BAILER DIMENSIONS:

TEST INFORMATION

TYPE: _____ TIME INTERVAL: _____
START TIME: _____ END STATIC WATER LEVEL: _____
END TIME: _____ MAX/MIN WATER LEVEL: _____
DATA FILE NAME: _____ TEST TIME-MAX/MIN: _____
TRANSDUCER DEPTH:

MANUAL TESTING

TIME (SEC)	DEPTH TO WATER (FT)	DEPTH TO DRAWDOWN	TIME (SEC)	DEPTH TO WATER (FT)	DEPTH TO DRAWDOWN
0					

COMMENTS:

SAMPLING RECORD: GROUNDWATER -

	CONSULTANT: PARSONS ES	WELL #:

PROJECT: _____
LOCATION: _____

DATE: _____
INSPECTORS: _____
PUMP #: _____
BLADDER #: _____

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS

MONITORING

INSTRUMENT	DECTECTOR

WELL DIAMETER FACTORS

DIAMETER (INCHES):	1	1.5	2	3	4	5	6	7	8	9	10
GALLONS / FOOT:	0.041	0.092	0.163	0.367	0.654	1.02	1.47	2.00	2.61	3.30	5.87
LITERS/FOOT	0.155	0.348	0.617	1.389	2.475	3.861	5.564	7.57	9.879	12.49	22.22

STANDING WATER VOLUME = WELL DIAMETER FACTOR * WATER COLUMN

HISTORIC DATA	DEPTH POW (TOC)	DEPTH TOP OF SCREEN	WELL DEV. TURBIDITY	WELL DEV. pH	WELL DEV. SPEC. COND

DATA COLLECTED AT WELL SITE	HEADSPACE READING	METALS TURBIDITY	VOLUME OF WATER ADDED TO HOLE (UNIT)	SAMPLE NUMBER(S)	SAMPLE INTERVAL

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)		

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	PUMPING RATE (L/min)	CUMULATIVE VOL (GALLONS/LITERS)	TEMP (C)	SPEC. COND (umhos)	DO (PPM)	pH	OXD-RED POTENTIAL Eh (mV)	TURBIDITY (NTU)

		Sample Result	Duplicate Result				
Field Analysis:							
Field Analysis:							
Field Analysis:							
Field Analysis:							
Field Analysis:							
Field Analysis:							
Field Analysis:							

SAMPLING RECORD: GROUNDWATER - GEOPROBE

SIKORSKY AIRCRAFT	CONSULTANT: PARSONS ES	WELL #:

SAMPLING ORDER		APPX IX	TAL/ TCL	PRESERV.	BOTTLES COUNT/ VOLUME	BOTTLES TYPE	SAMPLE NUMBER	TIME	CHECKED BY/ DATE
1	VOA								
1A	8010/8020								
2	SVOC								
2A	TPH								
3	HERB								
4	PEST/PCB								
5	DIOX./FURAN								
6	METALS + Sn	TAL							
7	CN								
8									
9									
10									
11									
12									
13	ANIONS								
14									
15									

COMMENTS: BOTTLE COUNTS ARE TRIPLED IF MS/MSD SAMPLES ARE COLLECTED

* Justification for Filtration:

SAMPLE LABELS:

VEGETABLE OIL INJECTION REPORT

Parsons ES		WELL #:

PROJECT : _____
LOCATION: _____

DATE: _____
PROJECT NO. : _____

DRILLING METHOD (s): _____
PUMP METHOD (s): _____
SURGE METHOD (s): _____
INSTALLATION DATE: _____
DEVELOPMENT DATE: _____

INSPECTOR: _____
CONTRACTOR: _____
CREW: _____
INJECTION DATE: _____

WATER DEPTH (TOC): _____ ft
WELL DIA. (ID CASING): _____ ft
BORING DIAMETER: _____ ft

INSTALLED POW DEPTH(TOC): _____ ft

DIAMETER FACTORS (GAL/FT):

DIAMETER (IN):	2	3	4	5	6	7	8	9	10	11	12
GALLONS/ FT:	0.163	0.367	0.654	1.02	1.47	2.00	2.61	3.30	4.08	4.93	5.87

CASING VOLUME INSIDE WELL = WATER COLUMN X WELL DIAMETER FACTOR

_____ GAL.

STANDING WATER IN SAND PACK =
WATER COL. BELOW SEAL(ft) X (BORING DIAM. FACTOR - WELL DIAM. FACTOR) X 0.3

_____ GAL.

SINGLE STANDING WATER VOLUME = A + B =

_____ GAL.

ACTIVITY	START TIME	END TIME	ELAPSED TIME	Injection Material/Tracers	Volume Injected (Gal) Oil	Volume Injected (Gal) Water	Injection Pressure	Injection Rate	Coments
TOTALS/FINAL									

COMMENTS:

APPENDIX D.

QUALITY ASSURANCE PROJECT PLAN

FINAL

QUALITY ASSURANCE PROJECT PLAN FOR FIELD APPLICATION TO ENHANCE IN-SITU BIOREMEDIATION OF CHLORINATED SOLVENTS VIA VEGETABLE OIL INJECTION AT THE NAVAL INDUSTRIAL RESERVE ORDNANCE PLANT (NIROP), FRIDLEY, MINNESOTA

SEPTEMBER 17, 2001

REVISION 4

Prepared for:

NAVAL FACILITIES ENGINEERING COMMAND
SOUTHERN DIVISION
NORTH CHARLESTON, SOUTH CAROLINA

Prepared by:

Parsons Engineering Science, Inc.
1700 Broadway, Suite 900
Denver, Colorado 80290

Approvals:

Parsons ES Project Manager	Date
Parsons ES QA Officer	Date
Department of the Navy Project Manager	Date
CH2M Hill Project Manager	09/20/01 Date
EnChem, Inc. QA Manager	Date
U.S.EPA Region 5 Remedial Project Manager	Date
U.S.EPA Region 5 Quality Assurance Reviewer	Date

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ACRONYMS AND ABBREVIATIONS

%D	Percent difference
%R	Percent recovery
θC	Degrees Celsius
Πs/cm	Microsiemans per centimeter
µg/kg	Microgram per kilogram
µg/L	Microgram per liter
A2LA	American Association Of Laboratory Accreditation
ANSI	American National Standards Institute
ASQC	American Society Of Quality Control
BFB	4-Bromofluorobenzene
CCAL	Continuing calibration
CCB	Continuing calibration blank
CCC	Continuing calibration check
CCS	Contract compliance screening
CCV	Continuing calibration verification
CF	Control factor
CFR	Code Of Federal Regulations
COC	Chain Of Custody
DCA	Dichloroethane
DCB	Dichlorobenzene
DCE	Dichloroethene
DD	Decision document
DF	Dilution factor
DQA	Data quality assessment
DQO	Data quality objective
EDB	1,2-dibromoethane
EDD	Electronic data deliverable
EICP	Extracted ion current profile
EPA	United States Environmental Protection Agency
FID	Flame ionization detector
G	Glass
GC/MS	Gas chromatograph/mass spectrometer
gm	Gram
HCl	Hydrochloric acid
H2SO4	Sulfuric acid
ICAL	Initial calibration
ICB	Initial calibration blank
ICV	Initial calibration verification
IDL	Instrument detection limit
IS	Internal standard
LCL	Lower control limit
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
MB	Method blank

ACRONYMS AND ABBREVIATIONS (Continued)

MDL	Method detection limit
mg/kg	Milligram per kilogram
mg/L	Milligram per liter
ml	Milliliter
MS/MSD	Matrix spike/matrix spike duplicate
Na2S2O3	Sodium thiosulfate
NBS	National Bureau Of Standards
NFG	National Functional Guidelines
NIROP	Naval Industrial Reserve Ordnance Plant
NIST	National Institute Of Standards And Technology
P	Polyethylene
PARCC	Precision, accuracy, representativeness, comparability, and completeness
Parsons ES	Parsons Engineering Sciences, Inc.
PC	Percent complete
PID	Photo-ionization detector
ppb	Parts per billion
PQL	Practical quantitation limit
PRL	Project reporting limit
QA	Quality assurance
QAPP	Quality assurance project plan
QC	Quality control
r	Correlation coefficient
r2	Coefficient of determination
RBC	Risk-based concentration
RF	Response factor
RPD	Relative percent difference
RRF	Relative response factor
RSD	Relative standard deviation
RT	Retention time
RTW	Retention time window
SD	Sample duplicate
SDG	Sample delivery group
SOP	Standard operating procedure
SPCC	System performance check compound
SQL	Sample quantitation limit
T	Brass sleeves in sample barrel
TCA	Trichloroethane
TCE	Trichloroethene
TIC	Tentatively identified compound
TOC	Total organic carbon
UCL	Upper control limit
VOC	Volatile organic compound

DISTRIBUTION LIST

Name	Affiliation	Signature	Date
Joel Sanders	Southern Division Naval facilities Command		
Venky Venkatesh	CH2M Hill Constructors		
R. W. John Aubert	Naval Warfare Assessment Station, Gage Laboratory		
Alan E. Dietrich	Naval Warfare Assessment Station, Gage Laboratory		
Mark Conti	Code 90 AMC Engineering Field Activity, Mid-west		
Thomas Bloom	U.S. EPA Region 5		
L. Finkelberg	U.S. EPA Region 5		
David Douglas	Minnesota Pollution Control Agency		
Michael Convery	MN Department of Health, Well Management Section		
Jeff Perry	Anoka County Parks		
Laura Schmidt	Anoka County Parks		
Richard Kuhlthau	Techlaw		
Mark Sladic	Tetra Tech NUS, Inc.		

SECTION 1

PROJECT MANAGEMENT

This Quality Assurance Project Plan (QAPP) has been developed for use in conjunction with sampling activities for the in-situ bioremediation of chlorinated solvents via vegetable oil injection at the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota to ensure appropriate sample collection and analysis.

This document outlines the objectives and quality assurance and quality control (QA/QC) activities necessary to achieve the desired data quality goals.

This QAPP has been prepared using the following documents as guidance:

- U.S. Environmental Protection Agency (EPA), 1999, EPA Requirements for Quality Assurance Project Plans. EPA QA/R-5, Washington D.C. November 1999.
- Region 5, U.S. Environmental Protection Agency (EPA), 2000, Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan. Revision 0, June.
- EPA, 1994, Guidance for the Data Quality Objectives Process. EPA QA/G-4. Washington, D.C. September 1994.
- EPA. 1994 Contract Laboratory Program National Functional Guidelines for Organic Data Review.
- EPA. 1994 Contract Laboratory Program National Functional Guidelines for Inorganic Data Review.
- EPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Third Edition, SW 846 (November 1986, 1995 update).
- American National Standards Institute/American Society of Quality Control (ANSI/ASQC E-4-1994), "Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs," July 1994, (Draft).

1.1 PROJECT ORGANIZATION

Quality is as important as cost and schedule, and therefore receives an equal amount of management attention and effort. The primary responsibility for meeting the quality objectives of a project remains with the operational personnel. The QA staff is responsible for achievement and documentation of product or service quality. The QA staff is charged with verifying the achievement of quality as well as providing assistance to the project organization in developing and implementing viable QA methods. They will perform their work in accordance with the standards of their profession, accepted practices, and applicable regulations. In the absence of specific guidelines, they will

follow best scientific or technical judgment. The project organization chart is presented on Figure 1.1.

Regularly scheduled briefings will be conducted with both regulatory and AFCEE personnel. These briefings will aid in defining project goals and scope as well as redefining objectives, as needed, and addressing current issues as the projects progress. These meetings will allow for the coordination of field work, sampling and analysis, and feedback to contractors regarding what risks are indicated by the data.

1.1.1 Management Responsibilities

1.1.1.1 Project Manager/Technical Manager/Principle Investigator – Todd Wiedemeier

Responsibility for contract management activities and direct liaison with CH2M Hill. Prepares and negotiates contract modifications and fulfills CH2M Hill subcontract requirements. Responsibility for completing the requirements of the statement of work and for day-to-day communications with CH2M Hill and Parsons ES staff. Organizes personnel and resources to accomplish individual tasks. Responsible for the following specific activities under this work order:

- Tracking, scheduling, and reviewing project deliverables (work plans and results reports) authored by task managers to insure proper QA/QC is performed and deliverables are produced in a timely manner.
- Develops the scope of work and organizational approach to complete the work.
- Provide technical direction and management for technical protocols, all work scopes, data compilation, and results reports.
- Reviews all project deliverables. Directs correspondence and interacts with technical resources.

1.1.1.2 U.S. EPA Region 5 Remedial Project Manager - Thomas Bloom

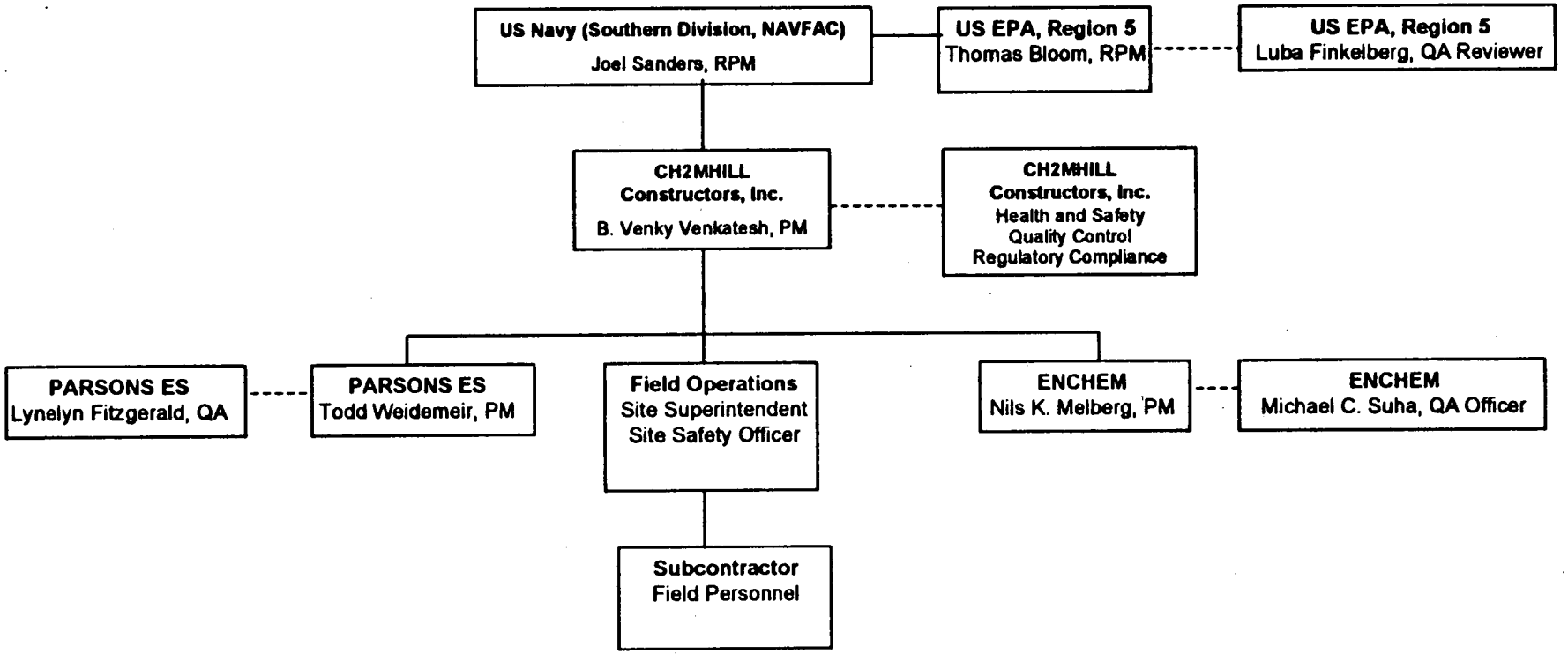
The EPA Remedial Project is responsible for directing and/or overseeing and coordinating all project activities. He is responsible for submitting QAPP and QAPP revisions and amendments to appropriate personnel within EPA Region 5 for review and approval.

1.1.1.3 CH2M Hill Project Manager - Venky Venkatesh

Responsible for the following specific activities under this project:

- Tracking and scheduling all project deliverables and field work,
- Management and technical direction of all work performed by subcontractors,

FIGURE 1.1 PROJECT ORGANIZATION CHART
NIROP FRIDLEY, MN
ANOKA COUNTY PARK VEGETABLE OIL INJECTION PILOT-SCALE STUDY



- Communication and coordination with Navy RPM, U.S. EPA Region 5 RPM, and MPCA staff,
- Review subcontractor performance,
- Select field personnel and resources,
- Review all project deliverables
- Prepare and submit status reports to Navy, U.S EPA Region 5, and MPCA.

1.1.2 QA Responsibilities

1.1.2.1 Parsons ES Quality Assurance Officer – Lynelyn Fitzgerald

The Project QA Officer is responsible for ensuring that sufficient QA procedures are developed for the project, that adequate quality controls are imposed to achieve the required level of QC, that audits are conducted, if necessary, to verify the level of quality, and that these procedures and controls are implemented properly. Also is responsible for review and approval the project QAPP and completion of the data validation and data quality assessment tasks. The Project QA Officer coordinates directly with the PM.

1.1.2.2 U.S.EPA Region 5 Quality Assurance Reviewer – Luba Finfelberg

- Reviewing and approving the QAPP.
- Conducting external Performance and System Audits of laboratory and Field Activities.
- Reviewing and evaluating analytical field and laboratory procedures.

1.1.2.3 Peer Review – Bruce Henry

- Provides technical guidance and oversight to meet the projects technical directives.
- Reviews project deliverables for technical accuracy.

1.1.3 Field Responsibilities - Parsons Field Oversite Manager – Dan Griffiths

Assists the Project Manager and Technical Director in day to day organization and execution of the enhanced bioremediation field application test with the following specific responsibilities:

- Assists project manager in tracking and scheduling project deliverables and with communications with client.
- Reviews/approves monthly invoices and monthly progress reports.

- Participates in a site visit to select well locations.
- Provides the primary line of communication with CH2M Hill field personnel.
- Provides the project manager with timely updates on task progress.
- Provides oversight of the field team in well installation, vegetable oil injection, and performance monitoring. Communicates progress and concerns to the project manager and technical director.
- Tracking of field and analytical data to insure report databases are complete.
- Identifies and documents nonconformance problems and subsequent corrective actions.

1.1.4 Field Personnel

Field personnel will include CH2M Hill Site Superintendent, Site Safety Officer, Site Quality Control Officer, and subcontractor staff with the following specific responsibilities:

- Responsible for all site activities;
- Provide direction to subcontractor personnel;
- Provide daily status reports to project manager;
- Conduct and participate in daily safety meeting;
- Stop work for unsafe condition or practices.

1.1.5 Laboratory Responsibilities

Enchem Inc. will be employed as the fixed based laboratory for sample analyses performed under this project. Enchem Inc. must signify acceptance of this QAPP with the appropriate Enchem project managers signature on the front cover. The signed QAPP will be submitted to CH2M Hill.

1.1.5.1 Laboratory QA Officer – Michael C. Suha

The Laboratory QA Officer is responsible for ensuring that sufficient QA procedures are applied to laboratory analyses. The Laboratory QA Officer is also responsible for ensuring that adequate laboratory controls are utilized for a high level of data quality, and that data program data quality objectives (DQOs) are met. Responsibilities of the Laboratory QA Officer include the following:

- Initiating nonconformance reports and/or corrective actions as necessary;
- Verifying the completion of corrective actions for major nonconformances cited in audits;

- Reviewing all statistical data to verify that the analytical laboratories are meeting stated QC goals; and
- Coordinating with the project chemist and Laboratory PM, and participating in system audits.

1.1.5.2 Laboratory PM – Nils K. Melberg

The Laboratory PM is responsible for implementation of the QAPP and laboratory subcontract at the laboratory level. The Laboratory PM ensures that project-required QA/QC procedures are specified for field and laboratory activities, and works directly with field personnel and the contractor's PM to ensure that procedures are adequate for the project-specified level of data quality. The Laboratory PM acts as the primary point of contact between the subcontract laboratory and the contractor.

1.2 PROBLEM DEFINITION/BACKGROUND INFORMATION

This project is being conducted by Parsons ES and Southern Division, Naval Facilities Engineering Command to document the enhanced bioremediation of chlorinated solvents dissolved in groundwater and sorbed to the aquifer matrix by injecting vegetable oil into the subsurface below the water table. Specifically, the objective of this field application is to determine if vegetable oil injection is a viable treatment option for VOCs in groundwater at the NIROP facility and adjacent areas including the Anoka County Park.

The chronological history of the site, existing site conditions, past and current chemical uses information, rationale for inclusion of chemical and non-chemical analyses, and site maps are described in detail in the Work Plan and other project documents. Table 1.1 provides a cross-reference to the document, date, section and page numbers for each QAPP requirement considered necessary to this section.

TABLE 1.1
CROSS-REFERENCE TABLE FOR QAPP ELEMENTS IN OTHER PROJECT DOCUMENTS
NIROP
FRIDLEY, MINNESOTA

QAPP Requirement	Document Title	Date	Section No.	Page(s)
Chronological History of the Site	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	1.2	1-3
Existing Site Conditions	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	2.2	2-7 to 2-9
Past and Current Chemical Use Information	Field Investigation Report at the NIROP and Anoka County Riverfront Park, Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	April 2000	1.0	1-1 to 1-3
Rationale for Inclusion of Chemical and Non-chemical Analyses	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	2.2 3.1	2-7 3-1
Site Maps	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	Figure 1.1 Figure 4.1	1-2 4-8
Field Methods	Work Plan for Field Application to Enhance *In-Situ*	May 2001	Table 4.4	4-13

QAPP Requirement	Document Title	Date	Section No.	Page(s)
	Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota			
Fixed Base Laboratory Methods	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	Table 4.4	4-13
Rationale for Sampling Matrices, Concentration Levels, and Analytical Parameters	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	3.0	3-1 to 3-4
Sampling Rationale	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	4.3.1	4-6
Project Documents	Work Plan for Field Application to Enhance *In-Situ* Bio-remediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, MN	May 2001	Section 7.0	7-1
Sample collection Records/Reporting	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	4.4.5	4-18 to 4-20

QAPP Requirement	Document Title	Date	Section No.	Page(s)
QC Sample Records/Reporting	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	4.4.5 5.1	4-18 to 4-20 5-1 to 5-3
Field Analysis Records/Reporting	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	4.4.5 5.1	4-18 to 4-20 5-1 to 5-3
Sampling Procedures	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	4.4.5	4-18 to 4-20
Cleaning and Decontamination of Equipment and Samples	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	4.3.3.6 4.4.2.1	4-11 4-14
Field Equipment Maintenance, Testing, and Inspection Requirements	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	4.4.2	4-14 to 4-15
Filing Structure	Project Management Plan Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval		Appendix E	E-1

QAPP Requirement	Document Title	Date	Section No.	Page(s)
	Industrial Reserve Ordnance Plant, Fridley, Minnesota			
Laboratory SOPs	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	Appendix F	F-1
Quantity and Types of QC Samples	Work Plan for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant, Fridley, Minnesota	May 2001	Table 5.1	5-2

1.3 PROJECT DESCRIPTION AND SCHEDULE

The sampling program for groundwater is designed to establish a baseline level of contaminants and then monitor the in-situ bioremediation of chlorinated solvents after injection of vegetable oil. Field activities will include installation and sampling of groundwater from newly installed monitoring wells, and soil sample collection for analysis of volatile organic compounds (VOCs) and total organic carbon (TOC).

Site-specific activities in support of the enhanced bioremediation field application will include:

- Installation of 1 new background monitoring well in the vicinity of existing monitoring well MS-46S, 3 vegetable oil injection wells, 9 groundwater monitoring wells, and 3 contingency monitoring wells;
- Baseline (i.e., pre-injection) sampling of groundwater at the newly installed injection wells, groundwater monitoring wells, contingency monitoring wells, and existing wells MS-46S, 27S, and MS-47S in accordance with the Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater (U.S. Environmental Protection Agency [USEPA], 1998);
- Pre-injection aquifer testing (hydraulic conductivity) of the 3 injection wells and 6 monitoring wells;
- Plumbing of the injection wells and injection of up to 3,600 gallons of food-grade vegetable oil (1,200 gallons per well);
- Post-injection aquifer testing (hydraulic conductivity) of the previously-tested 3 injection wells;
- Surveying of the newly installed injection and monitoring wells;
- Post-injection sampling of groundwater and vegetable oil (if present) at the newly installed monitoring and injection wells, in accordance with the Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater (USEPA, 1998) at 2, 5, 8, and 12 months after injection; and
- Preparation of a report detailing the results of the field application.

A list of the project analytical methods by matrix is presented in Table 1.2. The target compound list per analytical method with applicable target project quantitation limits, laboratory method detection limits (MDLS) and MCL are presented in Table 1.3. Target reporting limits were chosen based on risk-based target concentrations. The DQOs for this project have been finalized based on discussions with USEPA, MPCA, and Navy. Attachment 2 includes a discussion of the project-specific DQOs.

The following information is presented in the Work Plan (Table 1.1 provides the specific reference):

- ξ The rationale for sampling matrices, concentration levels, and analytical parameters;
- ξ The rationale for the sampling;

TABLE 1.2
FIXED-BASE LABORATORY ANALYTICAL METHODS
NIROP
FRIDLEY, MINNESOTA

Parameter	Analytical Method
Groundwater:	
VOCs	SW8260B
Methane/Ethene/Ethane	5021
Total Organic Carbon	E415.1
Nitrate/Nitrite	E300.1
Hydrogen[a/]	AM-20-GAX (modified SW8015)
Soils	
VOCs	SW8260B
Total Organic Carbon	SW846 Method 9060
Bulk Density	ASTM D2973
Vegetable Oil	
VOCs	SW8260B

Acronyms:

E - Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020. (EPA, 1983)
SW - Test Methods for Evaluating Solid Wastes, SW846, Third Edition, (EPA, 1995b)
VOCs – Volatile organic compounds
[a/] Test performed at Microseeps Laboratories. All other analyses will be performed by Enchem Laboratories.

TABLE 1.3
PROJECT QUANTITATION LIMITS, LABORATORY METHOD DETECTION LIMITS AND MCLs
NIROP
FRIDLEY, MINNESOTA

		Water				Vegetable Oil			Soil		
Parameter/ Method	**Analyte**	**MCL**	**Lab MDL[1/]**	**PQL[2/]**	**Units**	**Lab MDL[1/]**	**PQL[2/]**	**Units**	**Lab MDL[1/]**	**PQL[2/]**	**Units**
VOCs	1,1,1-TCA	200	0.53	1.0	µg/L	1.03	5.0	µg/kg	1.03	5.0	µg/kg
SW8260B	1,1,2,2-Tetrachloroethane		0.49	1.0	µg/L	0.83	5.0	µg/kg	0.83	5.0	µg/kg
	1,1,2-TCA	5	0.44	1.0	µg/L	1.22	5.0	µg/kg	1.22	5.0	µg/kg
	1,1-DCA		0.47	1.0	µg/L	1.05	5.0	µg/kg	1.05	5.0	µg/kg
	1,1-DCE	7	0.47	1.0	µg/L	1.24	5.0	µg/kg	1.24	5.0	µg/kg
	1,2-DCA	5	0.52	1.0	µg/L	1.21	5.0	µg/kg	1.21	5.0	µg/kg
	cis-1,2-DCE		0.99	1.0	µg/L	1.10	5.0	µg/kg	1.10	5.0	µg/kg
	Trans-1,2-DCE		0.38	1.0	µg/L	1.11	5.0	µg/kg	1.11	5.0	µg/kg
	1,2-Dichloropropane	5	0.46	1.0	µg/L	0.87	5.0	µg/kg	0.87	5.0	µg/kg
	2-Hexanone		0.80	5.0	µg/L	1.93	10.0	µg/kg	1.93	10.0	µg/kg
	4-Methyl-2-pentanone		0.90	5.0	µg/L	1.79	10.0	µg/kg	1.79	10.0	µg/kg
	Acetone	700	1.64	5.0	µg/L	5.54	10.0	µg/kg	5.54	10.0	µg/kg

		Water				Vegetable Oil			Soil		
Parameter/ Method	Analyte	MCL	Lab MDL[1/]	PQL[2/]	Units	Lab MDL[1/]	PQL[2/]	Units	Lab MDL[1/]	PQL[2/]	Units
	Benzene	5	0.44	1.0	µg/L	0.94	5.0	µg/kg	0.94	5.0	µg/kg
	Bromodichloromethane	80	0.42	1.0	µg/L	0.95	5.0	µg/kg	0.95	5.0	µg/kg
	Bromoform	80	0.52	1.0	µg/L	0.85	5.0	µg/kg	0.85	5.0	µg/kg
	Bromomethane		0.69	2.0	µg/L	1.14	10.0	µg/kg	1.14	10.0	µg/kg
	2-Butanone		1.03	5.0	µg/L	1.56	10.0	µg/kg	1.56	10.0	µg/kg
	Carbon Disulfide		0.51	1.0	µg/L	1.18	5.0	µg/kg	1.18	5.0	µg/kg
	Carbon tetrachloride	5	0.54	1.0	µg/L	1.21	5.0	µg/kg	1.21	5.0	µg/kg
	Chlorobenzene	100	0.48	1.0	µg/L	0.77	5.0	µg/kg	0.77	5.0	µg/kg
	Chloroethane		0.69	2.0	µg/L	1.97	10.0	µg/kg	1.97	10.0	µg/kg
	Chloroform	80	0.47	1.0	µg/L	1.07	5.0	µg/kg	1.07	5.0	µg/kg
	Chloromethane		0.83	2.0	µg/L	1.20	10.0	µg/kg	1.20	10.0	µg/kg
	Dibromochloromethane	80	0.51	1.0	µg/L	0.84	5.0	µg/kg	0.84	5.0	µg/kg
	Ethylbenzene	700	0.39	1.0	µg/L	2.15	5.0	µg/kg	2.15	5.0	µg/kg
	Methylene chloride	5	0.69	1.0	µg/L	0.99	5.0	µg/kg	0.99	5.0	µg/kg

		Water				Vegetable Oil			Soil		
Parameter/ Method	**Analyte**	**MCL**	**Lab MDL[1/]**	**PQL[2/]**	**Units**	**Lab MDL[1/]**	**PQL[2/]**	**Units**	**Lab MDL[1/]**	**PQL[2/]**	**Units**
	Xylenes (Total)	10000	0.71	5.0	µg/L	2.69	15.0	µg/kg	2.69	15.0	µg/kg
	Styrene	100	0.24	1.0	µg/L	0.97	5.0	µg/kg	0.97	5.0	µg/kg
	TCE	5	0.44	1.0	µg/L	0.93	5.0	µg/kg	0.93	5.0	µg/kg
	Tetrachloroethene	5	0.39	1.0	µg/L	1.14	5.0	µg/kg	1.14	5.0	µg/kg
	Toluene	1000	0.42	1.0	µg/L	1.28	5.0	µg/kg	1.28	5.0	µg/kg
	Trans-1,3-Dichloropropene		0.39	1.0	µg/L	1.07	5.0	µg/kg	1.07	5.0	µg/kg
	Vinyl chloride	2	0.17	2.0	µg/L	1.89	10.0	µg/kg	1.89	10.0	µg/kg
SW5021	Methane		2.00	2.8	µg/L	NA	NA	NA	NA	NA	NA
	Ethane		1.60	5.6	µg/L	NA	NA	NA	NA	NA	NA
	Ethene		1.40	5.0	µg/L	NA	NA	NA	NA	NA	NA
E415.1	TOC		91.00	500.0	µg/L	NA	NA	NA	NA	NA	NA
SW 846 Method 9060	TOC		NA	NA	NA	NA	NA	NA	91	500	µg/kg
E300.1	Nitrate		0.15	0.20	µg/L	NA	NA	NA	NA	NA	NA
	Nitrite		0.13	0.20	µg/L	NA	NA	NA	NA	NA	NA

		Water				Vegetable Oil			Soil		
Parameter/ Method	**Analyte**	**MCL**	**Lab MDL[1/]**	**PQL[2/]**	**Units**	**Lab MDL[1/]**	**PQL[2/]**	**Units**	**Lab MDL[1/]**	**PQL[2/]**	**Units**
AM-20-GAX (modified SW8015)	Hydrogen		0.01	0.02	PPMV	NA	NA	NA	NA	NA	NA

1/ Method detection limits provided by EnChem for analytes except hydrogen.
2/ Quantitation limits provided by EnChem were raised to accommodate a minimum of 3X multiplier between the MDL and PRL.
3/ Project specific accuracy and precision standards are presented in the laboratory SOPs and are summarized in Attachment 3 provided by Enchem, Inc.

Acronyms:
DCA – Dichloroethane
DCE – Dichloroethene
MCL – Maximum contaminant level
mg/kg – Milligrams per kilogram
mg/L - Milligrams per liter
NA – not applicable
PPMV – parts per million by volume
PQL – Practical quantitation limit
VOCs – Volatile organic compounds
TCA – Trichloroethane
TCE - Trichloroethene
µg/kg – micrograms per kilogram
µg/L – micrograms per liter

- Fixed-base laboratory and field analyses;
- New or innovative techniques;
- Specialized equipment or analysis;
- Project documents;

The laboratory review of definitive data is a four-step process involving an evaluation by the analyst, a peer review, an administrative review, and a QA review. The contractor data review process is performed in two phases. The initial phase, contract compliance screening (CCS), consists of inspecting the laboratory data deliverables to determine if the contract requirements were met. The second phase, data validation, includes a review of data results to assess data usability and application of data qualifiers to the analytical results based on adherence to method protocols and laboratory-specific QA/QC limits. Method SW8260B will undergo data validation.

Quality assurance assessments will not be performed during this scope of work unless nonconformance problems or systematic errors become apparent during routine quality assurance activities.

The contractor will assess the usability of analytical data. Any limitations on data use will be expressed quantitatively to the extent practicable. This data usability review will include a review of the analytical methods, quantitation limits, and other factors important in determining the PARCC parameters.

The schedule for the current project is as follows:

- Present conceptual design to reviewers – February 1, 2001.
- Submit draft work plan to reviewers – February 28, 2001
- Reviewers submit comments on draft work plan – March 26, 2001.
- Submit response to comments and final work plan revisions – approximately one month or less after receiving comments on draft work plan.
- Finalize pilot test well locations – After groundwater levels have stabilized following startup of new extraction wells in April 2001.
- Initiate field work – Anticipated in June 2001 (after regulatory approval of the work plan and QAPP).
- Submit Results Report

The project was initiated on January 18, 2001, and the period of performance ends on September 9, 2002.

1.4 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The data quality objectives (DQOs) for the analyses specified in this QAPP are described in the EPA, 1994a Guidance for the Data Quality Objectives Process, EPA QA/G-4. An effective QA program addresses DQOs for field sampling, field screening, and laboratory analytical methods. The contractor's field QA efforts will focus on ensuring that collected samples are representative of the conditions in the various environmental media at the time of sampling, and that field screening analyses are conducted in accordance with the QAPP and this work plan. Fixed-based laboratory QA efforts will be aimed primarily at ensuring that analytical procedures provide sufficient accuracy and precision to reliably quantify contaminant levels in environmental samples. The contract laboratory will also ensure that analyzed portions are representative of each sample, and that the results obtained from analysis of each sample are comparable to those obtained from analysis of other similar samples.

1.4.1 Analytical Data Quality Levels

The analytical levels for this project's DQOs will conform to the two EPA-defined categories of data. These data categories are defined below:

Screening Data with Definitive Confirmation - Screening data are generated by rapid, less precise methods of analysis with less rigorous sample preparation. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, instead of elaborate extraction/digestion and cleanup. Screening data provide analyte identification and quantification, although the quantification may be relatively imprecise. Screening data without associated confirmation data are not considered to be data of known quality. Results of field laboratory analyses conducted at NIROP will be considered screening-category data.

Definitive Data - Definitive data are generated using rigorous analytical methods, such as approved EPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., chromatograms, spectra, and digital values) in the form of hard-copy printouts or computer-generated electronic files. Data may be generated at the site or at an offsite location, as long as the QA/QC requirements are satisfied. For the data to be definitive, QA/QC requirements must be met in either case. Results of fixed-based laboratory analyses of samples collected at NIROP will be considered definitive data.

Screening data with definitive confirmation and definitive data quality levels will be used as indicated:

- Screening-level analyses will include field analyses for pH, temperature, and specific conductance, redox potential, dissolved oxygen, ferrous iron, manganese, hydrogen sulfide, sulfate, alkalinity, chloride, and carbon dioxide. Data collected for health and safety monitoring (if any) will also be screening level results.

- Definitive analyses will be used to satisfy the requirements for groundwater monitoring of natural attenuation. Definitive data acquired during the investigation

will be used to determine if vegetable oil injection is a viable treatment option for VOCs in groundwater.

100% of the data analyzed by the subcontracting laboratory is definitive. Field analyses (screening data) are not considered to be definitive data.

1.4.2 Integration of DQOs

The overall QA objectives for the evaluation must be appropriate to meet the project DQOs. The QA/QC program will provide the basic guidelines for evaluating the analytical results and field data for the site. These QA objectives are qualitative summaries of qualitative and quantitative analyses requested for the project to ensure that the planned quality and quantity of data are sufficient to support an evaluation of vegetable oil injection as a viable groundwater treatment technology. QA/QC is ensured through appropriate sample collection, preservation, and transport methods combined with an evaluation of laboratory analytical performance through the analysis of QC samples.

When analytical data fail to meet the required QA objectives, the technical report will discuss why the objectives were not met and any resultant effects on the project DQOs. Two major categories of noncompliance with QC requirements need to be considered:

- Requirements that are fully under the laboratory's control; and
- Requirements limited by the nature of the sample matrix.

Corrective action for noncompliance with QC standards that are fully under the laboratory's control (e.g., laboratory blanks, calibration standards, tuning, and laboratory check or control samples) will be addressed with a thorough reevaluation of the system and all calculations and, where practical, re-analysis of noncompliant samples. Corrective action for noncompliance with QC standards that are limited by the nature of the sample matrix (e.g., field blanks, matrix spikes, and duplicates) will be addressed with a thorough check of the system and all calculations, and the attachment of appropriate data qualifiers to noncompliant data.

An effective QA program addresses quality objectives for both sampling and laboratory methodologies. Parsons ES's field QA efforts are aimed primarily at assuring that samples are representative of the conditions in the various environmental media at the time of sampling. Laboratory QA efforts are aimed primarily at assuring that analytical procedures provide sufficient accuracy and precision to quantify contaminant levels in environmental samples. The Enchem laboratory Quality Assurance Manual (QAM) Plan is provided in Appendix E. The laboratory will also ensure that analyzed portions are representative of each sample, and that the results obtained from analysis of each sample are comparable to those obtained from analysis of other similar samples.

1.4.3 QA Objectives For Measurements

The QA objectives for all measurement data include considerations for precision, accuracy, completeness, representativeness and comparability (PARCC). These data quality assessment criteria will be used to evaluate the quality of the field sampling efforts, field-

screening results, and fixed-based laboratory results for compliance with project DQOs. Procedures used to assess data accuracy and precision are in accordance with the respective analytical methods from the EPA's (1995) Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW846.

1.4.3.1 Precision

Precision is the measure of variability among individual sample measurements under prescribed conditions. The relative percent difference (RPD) between field duplicate samples, laboratory sample duplicate (SD) pairs, and matrix spike/matrix spike duplicate (MS/MSD) sample results demonstrate the precision of the sample matrix. During collection of samples, precision can be affected by the spatial variability of pollutant concentrations. Collection of the of field duplicate samples, sample duplicate pairs and MS/MSD pairs will enable a determination of variability due to sampling and laboratory analysis practices. Because the concentration of analytes may be below detection limits in many environmental samples, RPD data will be generated by preparing matrix spikes in duplicate. The precision of the analytical method will thus be measured by calculating the RPD between the duplicate spikes, rather than environmental samples. Levels of precision will vary according to the sample matrix, the specific analytical method, and the analytical concentration relative to the method detection limit. For sample duplicate samples, the target RPDs will be based on the lab-established limits based on laboratory control charts. For field duplicate samples, the target RPDs are 25 percent for water and 35 percent soil samples. Project specific precision standards are presented in the laboratory standard operating procedures provided by Enchem, Inc.

When the laboratory control sample (LCS) results meet the accuracy criteria, results are also believed to be precise, and represent the precision of the laboratory independent from sample matrix. This is based on the LCS being within control limits in comparison to LCS results from previous analytical batches of similar methods and matrices.

Precision will be expressed in terms of RPD between the values resulting from primary and duplicate sample analyses. RPD is calculated as follows:

$$RPD = [(x1 - x2)/X][100]$$

where:

x1	=	analyte concentration in the primary sample,
x2	=	analyte concentration in the duplicate sample, and
X	=	average analyte concentration in the primary and the duplicate sample.

Acceptable levels of precision will vary according to the sample matrix, the specific analytical method, and the analytical concentration relative to the method detection limit (MDL). For field duplicate samples, the target RPDs are = 35 percent for soil and 25 percent water samples. Precision criteria for the laboratory QC samples must be defined by historical control limits developed through the use of control charts (Section 2.5.2.2).

An RPD within the control limit indicates satisfactory precision in a measurement system.

1.4.3.2 Accuracy

Accuracy is a measure of the closeness of a reported concentration to the true value. Accuracy is expressed as a bias (high or low) and is determined by calculating percent recovery (%R) from MS/MSD and surrogate spike recoveries indicate accuracy relevant to a unique sample matrix. LCS recoveries indicate accuracy relevant to an analytical batch lot, and are strictly a measure of analytical accuracy conditions independent of samples and matrices. The %R of an analyte, and the resulting degree of accuracy expected for the analysis of QC spiked samples, are dependent upon the sample matrix, method of analysis, and the compound or element being measured. The concentration of the analyte relative to the detection limit of the method is also a major factor in determining the accuracy of the measurement. Project specific accuracy standards are presented in the laboratory standard operating procedures provided by Enchem, Inc.

During field sampling and sample shipping, contamination may be introduced to the samples that could affect the accuracy of analysis results. Field and trip blanks will be used during sample collection and shipment to detect field contamination. Contamination affecting accuracy can also be introduced during laboratory analysis. Method blanks will be used during laboratory procedures to assess laboratory-introduced contamination. Laboratory results will be monitored, as they become available in order to identify potential sources of field or laboratory contamination, and eliminate them.

Accuracy expressed as %R is calculated as follows:

$$\%R = [(A-B)/C] \times 100$$

where:

A	=	spiked sample concentration,
B	=	measured sample concentration (without spike), and
C	=	concentration of spike added.

Accuracy criteria for the laboratory must be defined by historical control limits developed through the use of control charts (see Section 2.5.2.2).

Field measurements for parameters such as pH will be assessed for accuracy in the field. Specifically, field instruments will be assessed for accuracy by the response to a known calibration standard sample. The objective for accuracy of field measurements is to achieve and maintain factory QC specifications for the field equipment.

1.4.3.3 Completeness

Completeness is defined as the percentage of laboratory measurements judged to be valid on a method-by-method basis. Valid data are defined as all data and/or qualified data considered to meet the DQOs for this project. Data completeness is expressed as

percent complete (PC) and should be = 90 percent. The goal for meeting analytical holding times is 100 percent. At the end of each sampling event, the completeness of the data will be assessed. If any data omissions are apparent, the parameter in question will be resampled and/or reanalyzed, if feasible. The laboratory results will be monitored, as they become available to assess laboratory performance and its effect on data completeness requirements. When appropriate, additional samples will be collected to ensure that laboratory performance meets PC requirements.

PC is calculated as follows:

$$PC \quad \frac{N_A}{N_I} X100$$

Where:

N_A = Actual number of valid analytical results obtained, and

N_I = Theoretical number of results obtainable under ideal conditions.

1.4.3.4 Comparability

Comparability expresses the confidence with which data from one sample, sampling round, site, laboratory, or project can be compared to those from another. Comparability during sampling is dependent upon sampling program design and time periods. Comparability during analysis is dependent upon analytical methods, detection limits, laboratories, units of measure, and sample preparation procedures.

Comparability is determined on a qualitative rather than quantitative basis. For this project, comparability of all data collected will be ensured by adherence to standard sample collection procedures, standard field measurement procedures, and standard reporting methods, including consistent units. For example, concentrations will be reported in a manner consistent with general industry practice (e.g., soil data will be reported on a dry-weight basis).

In addition, to support the comparability of fixed-base laboratory analytical results with those obtained from previous or future testing, all samples will be analyzed by EPA-approved methods, where available. The EPA-recommended maximum permissible sample holding times for organic and inorganic parameters will not be exceeded. All analytical standards will be traceable to standard reference materials. Instrument calibrations will be performed in accordance with EPA method specifications, and will be checked at the frequency specified for the methods. The results of these analyses can then be compared to analyses by other laboratories and/or to analyses for other sites addressed by this site investigation.

1.4.3.5 Representativeness

Representativeness expresses the extent to which collected data define site contamination. Where appropriate, sample results will be statistically characterized to determine the degree to which the data accurately and precisely represent a characteristic

of a population, parameter variation at a sampling point, a process, or an environmental condition. Sample collection, handling, and analytical procedures are designed to obtain the most representative sample possible. The sample locations and procedures described in the Work Plan were designed with the consideration of obtaining samples representative of potentially contaminated areas. Sample handling and analytical procedures also incorporate consideration of obtaining the most representative sample possible. Representative samples will be achieved by the following:

- ξ Collection of samples from locations fully representing site conditions;
- ξ Use of appropriate sampling procedures, including proper equipment and equipment decontamination;
- ξ Use of appropriate analytical methods for the required parameters and project reporting limits; and
- ξ Analysis of samples within the required holding times.

The portion of each sample chosen for analysis also affects sample representativeness. The laboratory will adequately homogenize all samples prior to taking aliquots for analysis to ensure that the reported results are representative of the sample received. Because many homogenization techniques may cause loss of contaminants through volatilization, homogenization will not be performed on samples for VOC analyses.

The sample representativeness will be preserved by using correct field sample collection and handling procedures, properly decontaminating sampling equipment, and using field QC samples, where appropriate. Sample collection and equipment decontamination procedures are discussed in Section 4.0 of the Work Plan. These procedures will be followed to collect samples that are representative of onsite environmental conditions.

1.4.3.6 Sensitivity

The concentration of any one target compound that can be detected and/or quantified is a measure of sensitivity for that compound. Sensitivity is instrument-, compound-, method-, and matrix-specific. The definitions of terms relating to sensitivity and DQOs are presented in Section 2.5.2.4.

1.5 DOCUMENTATION AND RECORDS

The following documents and records are also presented in the Work Plan (Table 1.1 provides the specific reference):

- ξ Sample collection documentation
- ξ QC sample records,
- ξ Field analysis records,
- ξ Field data reporting

Data deliverables required for the analytical results include both a hard copy and an electronic copy. Hardcopy reporting of analytical results is defined in Table 1.4. The laboratory will be required to provide two copies of each hard copy data-reporting package.

To facilitate data handling and management, both field and laboratory data will be entered into a computerized format. All data entered into the electronic data files will correspond to the data contained in the original laboratory reports and other documents associated with sampling and the laboratory hard copy data deliverable packages. The subcontracting laboratories will provide all data and QC data in a standard electronic data deliverable (EDD) format that is provided as Attachment 1.

TABLE 1.4
REQUIRED HARDCOPY LABORATORY DELIVERABLES
NIROP
FRIDLEY, MINNESOTA

Method Requirements	Laboratory Deliverables (Definitive Data)
Requirements for all methods:	
Case narrative	Project identification
	Analytical method description and reference citation

TABLE 1.4 (Continued)
REQUIRED HARDCOPY LABORATORY DELIVERABLES
NIROP
FRIDLEY, MINNESOTA

Method Requirements	Laboratory Deliverables (Definitive Data)
Requirements for all methods:	
	Discussion of unusual circumstances, problems, and nonconformances
Monthly quality assurance (QA) report	Any format to discuss issues which may affect data quality
Chain-of-custody (COC) form	Signed and dated when samples were received at laboratory
Dates of sample preparation and analysis (including first run and subsequent runs).	Specific deliverable depends upon type of analysis
Quantitation limits achieved.	Specific deliverable depends upon type of analysis
Dilution or concentration factors.	Specific deliverable depends upon type of analysis
Summary analytical batch report including analytical batch samples, method of analysis, matrix description, date of sample collection and receipt, laboratory identification number of each environmental sample plus identification number of each batch quality control (QC) sample (including matrix spike/matrix spike duplicate (MS/MSD), calibration check, etc.).	Any format
Method reporting limits	QC summary report
QC limits	QC summary report
Corrective action reports.	Any format
Laboratory data validation/review checklists	Any format
Percent moisture for all soil samples	Any format
Requirements for organic analytical methods:	
Sample data sheets	Summary information only [a/]
Surrogate recoveries	Summary information only
MS/MSD	Summary information only

TABLE 1.4 (Continued)
REQUIRED HARDCOPY LABORATORY DELIVERABLES
NIROP
FRIDLEY, MINNESOTA

Method Requirements	Laboratory Deliverables (Definitive Data)
Requirements for organic analytical methods (Cont'd):	
Method blank analysis	Summary information only
Laboratory control spike (LCS)	Summary information only
Internal standard area and retention time summary data	Summary information only
Analysis run log	Any format
Requirements for inorganic analytical methods Metals:	
Sample data sheets	Summary information only *
Method blank, taken through sample preparation.	Summary information only
Laboratory control spike/laboratory control spike duplicate	Summary information only
Matrix spike/matrix spike duplicate	Summary information only
Post-digestion spike sample recovery	Summary information only
Method of standard additions	Summary information only
Analysis run logs	Any format

a\ Summarized results can be in any format that provides the necessary data to completely validate that QC parameter. Example formats are the forms equivalent to those defined for the EPA Contract Laboratory Program or SW846 programs.

SECTION 2

DATA GENERATION AND ACQUISITION

2.1 SAMPLING PROCESS DESIGN

Sampling rationale and site maps are described in detail in the Work Plan and other project documents. Table 1.1 provides a cross-reference to the document, date, section and page numbers for each QAPP requirement considered necessary to this section.

2.2 SAMPLING METHODS REQUIREMENTS

Sampling procedures are discussed in the Work Plan (see Table 1.1). These include:

- Cleaning and Decontamination of Equipment and Sample Containers
- Field Equipment Maintenance, Testing and Inspection Requirements
- Inspection and Acceptance Requirements for Supplies and Sample Containers

2.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

2.3.1 Field sample Handling and Custody Procedures

Sample custody begins in the field at the time of collection and continues throughout the laboratory analytical process. Proper sample custody procedures are needed to ensure that samples have been obtained from the locations stated and that they have reached the laboratory without alteration. All sample bottles shall be maintained onsite in a locked storage area prior to use. Evidence of the sample traceability from collection to shipment, laboratory receipt, and laboratory custody must be documented. A sample is considered to be in a person's custody if the sample is:

- In a person's actual possession,
- In view after being in a person's possession,
- Locked so that no one can tamper with it after having been in physical custody, or
- In a secured area, restricted to authorized personnel.

For samples to be submitted to the fixed-base laboratory, COC forms will be prepared at the time of sample collection and will accompany the samples through the laboratory sample processing. COC forms will be completed for each sample cooler for tracking purposes and to provide a written record of all persons handling the samples. Samples analyzed in the field will not require COC forms. The following information will be documented on the COC form for each fixed-base laboratory sample:

- Unique sample identification;

- Date and time of sample collection;
- Source of sample (including name, location, and sample type);
- Designation of MS/MSD;
- Preservative used;
- Analyses required;
- Name(s) of collector(s);
- Custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratory or laboratories; and
- Bill of lading or transporter tracking number (if applicable).

Shipments will be sent by common carrier for overnight delivery, and a bill of lading will be prepared. The shipping bill number will be recorded on the COC form. Bills of lading will be retained as part of the permanent project documentation and all sample shipments will be regulated by the USDOT as described in 49 CFR 171 through 177.

In general, all documents will be completed in permanent black ink. Errors will be corrected by crossing out with a single line and then dating and initialing the correction. The use of correction fluid is not permissible. The documents used during the field investigation will remain on site during the entire effort. Forms used will be organized in a central file also located on site.

2.3.2 Sample Numbering Conventions

All environmental samples collected as part of the vegetable oil injection pilot test will be numbered in strict accordance to the numbering scheme presented in this QAPP. All samples will bear sample numbers with the following format.

MATRIX/LOCATION ID/SAMPLING ROUND/SAMPLE CODE

For environmental samples where the matrix is soil, groundwater, or vegetable oil, the matrix identifier in the sample ID will consist of SL, GW, and VO respectively. The Location identifier portion of the sample ID will consist of the monitoring well, injection well, or soil boring where the sample was collected. In the instance where multiple samples are collected in a single location during a single sampling event, a number will be added to the location ID portion of the sample ID. The number will increase by one for each successive sample collected at the location (e.g. SL/MW01-1/1/SA, SL/MW01-2/1/SA, etc.). The sampling round portion of the sample ID will consist of a single digit which will represent which round of sampling the sample was collect from. The first round of sampling during installation of the pilot test well field will represent sampling round 1, and each successive sampling round following installation will bear a successively higher round number. The sample code portion of the sample ID will represent the type of sample. The sample code will consist of: SA for primary samples, TB for trip blanks, RB for rinsate blanks, EB for equipment blanks, MS for matrix spikes, and MSD for matrix spike duplicates. The sample code for field duplicate

samples will consist of SA in the sample code portion of the sample ID, but the location ID will consist of a nonexistent sampling location. The first field duplicate to be collected will bear the location ID MW900, and each successive field duplicate will be labeled with a successively higher location ID (e.g. MW900, MW901, etc.). All sampling information will be recorded in the field logbook at the time of sampling.

2.3.3 Laboratory Custody Procedures

To facilitate the documentation of sample custody, the laboratory will track the progress of sample preparation, analysis, and report preparation. When the laboratory receives the samples, custody information is checked against the samples received for discrepancies. Laboratory receipt and handling procedures are presented in detail below. Within one day of receipt of samples from the contractor, the laboratory will send signed facsimile copies of all COCs and sample log-in receipt forms to the contractor. All discrepancies and/or potential problems (e.g., lack of sample volume) will be discussed immediately with the contractor's project manager.

The laboratory sample custodian will be required to provide a report to the contractor of any problems observed with any of the samples received. This report will also document the condition of samples, sample numbers received, corresponding laboratory numbers, and the estimated date for completion of analysis. The laboratory must receive written permission from the contractor before sending any samples (originally scheduled to be analyzed at their facility) to another laboratory. Analysis will not be performed on samples whose integrity have been compromised or is suspect.

2.3.3.1 Sample Receipt and Handling

Laboratory sample custody will be maintained using the following procedures:

1. The laboratory will designate a sample custodian responsible for maintaining custody of the samples and all associated paperwork documenting that custody.

2. Upon receipt of the samples, the sample custodian will sign the original COC form and compare the analyses requested thereon with the label on each sample container.

3. A visual assessment of each sample container will be performed to note any anomalies such as broken or leaking bottles, lack of preservation (e.g., ice melted en route) or air bubbles in VOC sample bottles. VOC sample bottles should be shipped inverted. This assessment will be recorded as part of the incoming COC procedure.

4. If the COC and samples correlate, and there has been no tampering with the custody seals, the "received by laboratory" box on the COC form will be signed and dated.

5. Care will be exercised to document any labeling or descriptive errors. In the event of discrepancies, breakage, or conditions that could compromise the validity of analyses (i.e., cooler temperature), the laboratory project coordinator

will immediately contact the laboratory PM as part of the corrective action process. If there is a discrepancy on the COC form, the laboratory will call for resolution. If the form requires changes, the laboratory will make changes by drawing a single line through the item requiring correction and initial and date it. If additional information is added to the form, the laboratory will initial as date the changes and note "as per Project Manager". If the cooler temperature is above 4ρ2θC, the Project Chemist will be notified and the impact to data quality assessed. Samples will be appropriately qualified during the data validation review.

6. Samples will be logged into the laboratory management computer system, which includes a tracking system for extraction and analysis dates. The laboratory will assign a laboratory work number to each sample for identification purposes. The sample custodian will log the laboratory work number and the field sample identification into a laboratory sample custody log. The laboratory sample custody log may be either hard copy or computerized, depending on the laboratory's system.

7. The samples will be stored in a secured area at a temperature of approximately 4 ρ 2 degrees °C or cooler (as applicable) until analyses commence. The laboratory log should also contain the laboratory storage cooler number (if applicable) in which the sample will be stored while on the laboratory's premises. Samples will be logged when they are removed from and returned to storage for analysis. Samples must be stored in coolers separate from those used to store analytical standards, reagents, and/or QC samples.

8. The samples will be distributed to the appropriate analysts, with names of individuals who receive samples recorded in internal laboratory records.

9. The original COC form will accompany the laboratory report submittal to the contractor and will become a permanent part of the project records.

10. Data generated from the analysis of samples also will be kept under proper custody by the laboratory.

Upon analysis, a laboratory lot control number will be assigned to the sample. All samples within a given laboratory analysis group (e.g., samples sharing the same laboratory QC measurement samples) will have identical laboratory lot control numbers.

Disposal of sample containers and remaining sample material will be the responsibility of the laboratory. Samples should be disposed of appropriately when all analyses and related QA/QC work are completed.

2.3.3.2 Laboratory Sample Identification

The laboratory conducting the analysis of the samples will provide the data user with information on the laboratory sample identification system. With knowledge of this

laboratory sample identification system, data generated at the laboratory can be tracked by both the laboratory and field sample identification systems.

Each sample will be logged into the laboratory system by assigning it a unique sample number. This laboratory number and the field sample identification number will be recorded on the laboratory report.

2.3.3.3 Final Evidence Files

The file structure for this project is presented in the Project Management Plan. All data files will be maintained for seven years.

2.4 ANALYTICAL METHODS REQUIREMENTS

The analytical methods by matrix are presented in Table 1.2. SOPs for the fixed based laboratory methods are presented in Appendix E of the Work Plan (Table 1.1). The quantities and types of samples for each analyte are presented in the Work Plan (table 1.1).

2.5 QUALITY CONTROL REQUIREMENTS

2.5.1 Field Sampling Quality Control

Table 2.1 presents the instructions for field measurement calibration, maintenance and decontamination. Table 2.2 presents instructions for field measurement instrumentation, control parameters, control limits, and corrective actions.

TABLE 2.1
INSTRUCTIONS FOR FIELD MEASUREMENT INSTRUMENTATION CALIBRATION, MAINTENANCE, AND DECONTAMINATION
NIROP
FRIDLEY, MINNESOTA

Parameter	Equipment	Calibration	Source of Calibration Standards	Equipment Maintenance	Equipment Decontamination
pH (Water)	pH Meter, accurate to 0.01 pH units, beaker, extra batteries, and extra probe	Immediately before a well is purged use pH 7, and either pH 4 or pH 10 buffers depending on whether the pH is <7 or >7, respectively.	Commercially available, premixed solutions.	Store probe when not in use in pH 7 or pH 4 buffer solution; replace batteries as necessary.	Rinse probe with distilled water after each use and blot or shake to remove excess water.
Specific Conductance	Electrical conductance meter, beaker, extra batteries, and extra probe.	Daily according to manufacturer's instructions; use two standard µs/cm solutions that bracket expected range of values.	Commercially available, premixed solution.	Re-platinize probe according to manufacturer's instructions if readings become inaccurate; replace batteries as necessary.	Rinse probe with distilled water after each use and blot or shake to remove excess water.
Temperature	Mercury thermometer	Calibrated by manufacturer.	Not applicable.	Replace in case of malfunction.	Rinse tip with distilled water after each use and blot or shake to remove excess water.

TABLE 2.2
INSTRUCTIONS FOR FIELD MEASUREMENT INSTRUMENTATION CONTROL PARAMETERS, CONTROL LIMITS, AND CORRECTIVE ACTIONS
NIROP
FRIDLEY, MINNESOTA

Measurement Parameter	Control Checks	Control Limits	Corrective Actions (required if control limits are not achieved)
pH	Measure buffer pH at least following every other measurement.	Buffer measurements within ± 0.2 units of actual values.	Re-calibrate pH meter; check batteries and probe condition.
Specific conductance	Measure standard solution daily.	± 10 percent of actual value.	Replace or re-platinize probe; check temperature and adjust, if applicable; verify that meter zeros and redlines properly; check batteries.
Temperature	Check measurement.	± 1 degree Celsius.	Replace thermometer or correct temperature readings.

2.5.2 Analytical Quality Control Checks

Application of a specific analytical method depends on the sample matrix and the analytes to be identified. ENCHEM, Inc. of Green Bay, Wisconsin will be the fixed-base laboratory for this project. Standard operating procedures (SOPs), updated MDLs and reporting limits, and quality control limits will be obtained and kept on file for reference during validation and data assessment. ENCHEM. Inc SOPs are provided in Appendix F. Methods for each of the parameters likely to be included in the NIROP analytical program are EPA-approved. Analytical procedures will follow the established EPA methods wherever such methods exist for a specified analyte. All approved methods are presented in Table 1.2. The referenced methods are defined in EPA (1995) Test Methods for Evaluating Solid Waste, Physical and Chemical Methods, SW846, 3rd Edition, Update III and EPA (1983) Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020.

2.5.2.1 Field QC Samples (Analyzed by Fixed-Based Laboratory)

As a check on field sampling, QA/QC samples will be collected for all fixed-based laboratory analyses during each sampling event. Definitions of field QA/QC samples are presented below.

Field Duplicates/Replicates

A field duplicate or replicate is defined as two or more water (or soil) samples collected independently at the same sampling location during a single act of sampling. Soil samples are divided into two equal parts (replicates) for analysis. Field duplicates will be indistinguishable from other samples by the laboratory. Each of the field duplicates/replicates will be uniquely identified with a coded identifier in the same format as other sample identifiers. Duplicate sample results are used to assess the precision of the sample collection process. During the collection of VOC samples, compositing should not be performed because of the potential for target compound loss. Ten percent of all field samples will be field duplicates.

Trip Blanks

The trip blank is used to indicate potential contamination by VOCs during sample shipping and handling. A trip blank consists of analyte-free laboratory reagent water (δ18 μ ohm-cm deionized water) in a 40-milliliter (ml) glass vial sealed with a Teflon® septum. The blank accompanies the empty sample bottles to the field and is placed in each shipping cooler containing VOC samples returning to the laboratory for analysis. The trip blank is not opened until the corresponding site samples are analyzed. A trip blank is required to accompany all water samples collected for VOC analysis.

Equipment Rinsate Blank

Equipment rinsate blanks consist of (δ18 μ ohm-cm deionized water (or equivalent) poured into or pumped through the sampling device following decontamination. The rinsate is transferred to a sample bottle appropriate for the analysis and transported to the laboratory. The equipment rinsate samples are analyzed for the same laboratory parameters as the site samples. Equipment rinsate blanks are used to measure contamination introduced to a sample set from improperly decontaminated sampling equipment. Rinsate blanks will be collected daily on re-usable sampling equipment.

Temperature Blank

The temperature blank is used to indicate the temperature of the sample cooler upon receipt at the laboratory. A temperature blank consists of laboratory reagent in a 40-ml glass vial sealed with a Teflon® septum. Any cooler temperature exceeding the allowable 4 ρ 2 degrees Celsius (°C) must be noted and the project chemist notified prior to sample analyses. The impact to data quality assessed and a decision will be made to proceed with analysis or resample. If the laboratory proceeds with analysis, samples will be appropriately qualified during the data validation review.

2.5.2.2 Laboratory QC Samples and Analytical Requirements

Laboratory QC samples are necessary to determine the precision and accuracy of the analyses, confirm matrix interferences, and demonstrate target compound contamination of sample results. QC samples will be analyzed routinely by the analytical laboratory as part of the laboratory QC procedures. Contract laboratories performing definitive data quality analyses require a more stringent QC program than those performing screening-level data quality analyses do. Definitions of QC samples and analytical requirements are presented below. Control limits for MS, MSD and LCS samples represent historically established limits determined by control charts by the analytical laboratory. These control limits will be requested from the laboratory.

Holding Time

Holding times for sample extraction and/or analysis as required by the methods will be met for all samples. The holding time is calculated from the date and time of sample collection to the time of sample preparation and/or analysis. All sample analyses to include dilutions and second-column confirmation will meet the required holding times. Table 2.3 defines applicable method-specific analytical holding times.

Method Blanks

Method blanks are designed to detect contamination of the field samples in the laboratory environment. Method blanks verify that interferences caused by contaminants in solvents, reagents, glassware, or in other sample processing hardware are known and minimized. The method blank will be (δ18 μohm-cm deionized water (or equivalent) for water samples, and a purified solid matrix (Ottawa sand or equivalent) for soil samples. The concentration of target compounds in the blanks must be less than the project reporting limit (PRL). Exceptions are not made for common laboratory contaminants. If the blank contaminant concentration is not less than the specified limit, then the source of contamination will be identified, and corrective action will be taken. Sample quantitation limits (SQLs) and detection limits will not be raised because of blank contamination. Analytical data will not be corrected for the presence of analytes in blanks.

Laboratory Control Samples

LCSs are blank spikes made from clean laboratory-simulated matrices (reference method blank matrices) spiked with known concentrations of all target analytes of interest. The LCS is carried through the complete sample preparation and analysis procedure. LCSs are designed to check the instrument and method accuracy. An LCS will be analyzed with every analytical batch. Failure of the LCS to meet %R criteria requires corrective action before any further analyses can continue. All sample results associated with the out-of-control LCS must be re-extracted and reanalyzed after control has been re-established. All re-extraction and reanalysis must be performed within the sample holding times.

Surrogate Spike Analyses

Surrogate spike analyses are used to determine the efficiency of analyte recovery in sample preparation and analysis in relation to sample matrix. Calculated %R of the spike is used to measure the accuracy of the analytical method for an individual sample matrix. A surrogate spike is prepared by adding to an environmental sample (before extraction) a known concentration of a compound similar in type to the target analytes (i.e., a surrogate compound) to be analyzed. Surrogate compounds, as specified in the methods, will be added to all samples analyzed, including method blanks, MS/MSDs, LCSs, field samples, and duplicate samples. Failure of the surrogate to meet %R criteria requires corrective action. All sample results associated with the out-of-control surrogates must be reanalyzed. If the reanalysis does not provide an in control surrogate %R, the sample must be re-extracted and reanalyzed. All re-extraction and reanalysis must be performed within the sample holding time.

Matrix Spikes/Matrix Spike Duplicates

MS samples are designed to check the accuracy of the sample matrix (matrix bias) with respect to analytical procedures by analyzing a field sample spiked in the laboratory with a known standard solution containing all the target analytes. An MSD is the second of a pair of laboratory MS samples. The MSDs are designed to check the precision of sample matrix with respect to analytical procedures.

One MS/MSD pair will be collected for every group of 20 project samples of similar matrix. Field blanks or duplicates are not to be used as MS/MSDs. If surrogate and/or target compound concentrations are out of control in the MS or MSD, the out-of-control MS or MSD must be reanalyzed. If the reanalysis does not provide an in-control %R, and evidence of matrix interference is not apparent the MS/MSD pair must be re-extracted and reanalyzed. All re-extraction and reanalysis must be performed within the sample holding time. In cases where the concentration of a target compound in the parent sample is greater than four times the spike concentration of the same compound in the MS/MSD, no re-extraction or reanalysis is required. The MS/MSD results are considered unusable.

TABLE 2.3
REQUIREMENTS FOR CONTAINERS, PRESERVATION TECHNIQUES, SAMPLE VOLUMES, AND HOLDING TIMES
NIROP
FRIDLEY, MINNESOTA

Name	Matrix	Analytical Methods	Container	Preservation[a/]	Minimum Sample Volume or Weight	Maximum Holding Time
Nitrate/Nitrite	Water	E300.1	P, G	4°C	50 ml	48 hours
Total organic carbon	Water	E415.1	P, G	4°C, HCl or H_2SO_4 to pH < 2 DOC is filtered with glass fiber filters	500 ml	28 days
Total organic carbon	Soil	SW846 Method 9060	G, T	4°C	4 ounces	14 days
Methane/ Ethane/ Ethene	Water	SW5021	G, Teflon®- lined septum	4°C, HCl to pH < 2, 0.008% $Na_2S_2O_3$ [b/]	3 x 40 ml or 4 ounces	7 days
Volatile Organics	Water	SW8260B	G, Teflon®-lined septum	HCl to pH<2, 4°C, 0.008% $Na_2S_2O_3$ [b/]	3 x 40 ml	14 days
Volatile Organics	Soil	SW8260B	G, Teflon®-lined septum	4°C	4 ounces	14 days
Volatile Organics	Vegetable Oil	SW8260B	G, Teflon®-lined septum	4°C	3 x 40 ml	7 days
Hydrogen	Water	AM-20-GAX (modified SW8015)	1-Evacuated 22cc vial supplied by Microseeps	No preservation	22 cubic centimeters	14 days

a/ No pH adjustment for soil.

b/ Preservation with 0.008 percent $Na_2S_2O_3$ is only required when residual chlorine is present.

Acronyms:

cc – Cubic centimeter

G – Glass

HCl – Hydrochloric acid

H_2SO_4 – Sulfuric acid

$Na_2S_2O_3$ – Sodium thiosulfate (if residual chlorine is present)

P – Polyethylene

cc – cubic centimeter

T - brass sleeves in the sample barrel

Analytical Batches

Analytical batches will be designated in the laboratory at a minimum of one batch per sample delivery group (SDG). Each SDG will be composed of a maximum of 20 project samples of similar matrix collected within a 7-day period. Included in each SDG of 20 (or fewer) samples per analytical method will be an analytical batch identification number. This identification number will allow a reviewer to determine the association between field samples and QC samples. Analytical batches also will be inclusive of preparation lots (for methods with extraction processes) and calibration periods (for methods such as SW8260B where the extraction and analysis are simultaneous). The laboratory will, at a minimum, analyze internal QC samples at the frequency specified by the methods. These QC samples for each analytical batch include calibration standards and checks, blanks, an LCS, and a MS/MSD pair (SD and MS for inorganic analyses) per analytical batch.

Internal Standards

Internal standards (ISs) are compounds of known concentrations used to quantitate the concentrations of target detections in field and QC samples. ISs are added to all samples (analyzed for GC/MS methods only) after sample extraction or preparation. Because of this, ISs provide for the accurate quantitation of target detections by allowing for the effects of sample loss through extraction, purging, and/or matrix effects. ISs are used for any method requiring an IS calibration. Corrective action is required when ISs are out of control.

Control Limits

The acceptance criteria for the control limits associated with all methods will follow guidance established in the SW846 methods and the laboratory's historical data. The laboratory must specify historical accuracy and precision control limits for MS/MSDs, LCSs, and surrogate spikes for each analytical method presented in Table 1.2. The laboratory established limits must meet the DQOs for this project (Attachment 2). Each laboratory reviews and evaluates QC data through the use of method-specific control charts. At least 20 measurements are required before control limits can be established. Warning limits, when established, are set at two standard deviations above and below the mean standard recovery and are used by the laboratory as an indicator of potential impending analytical problems. Upper and lower control limits are defined as three standard deviations above or below the mean standard recovery, respectively, and are used to qualify data accordingly on the basis of out-of-control criteria. The control limits will be historic lab-established limits specific for that laboratory.

Control limits must be carefully reviewed by regulators and the contractor to ensure that the project DQOs will be met. Control limits established through the process described above can provide laboratory historical limits that will not satisfy project DQOs. Site-specific DQOs are presented in Attachment 2. For example, laboratory historical control limits with a low-end acceptability equivalent to the limit of detection (i.e., the MDL) are possible and not uncommon. Control limits at the limit of detection do not provide for acceptable data quality. In this instance, a re-evaluation of the control limits or an agreed minimum acceptable control limit may be required.

Calibration Requirements

Analytical instruments will be calibrated in accordance with the analytical methods and in accordance with the MDL as defined in Section 2.5.2.4. All analytes reported will be present in the initial and continuing calibrations, and these calibrations must meet the acceptance criteria specified in Table 2.4. The contract laboratory will maintain records of standard preparation and instrument calibration. Records will unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards will be traceable to standard materials.

Analyte concentrations are determined with either calibration curves (linear regression) or response factors (RFs). Calibration curves will be evaluated using a coefficient of determination (r^2), of greater than 0.990 or correlation coefficient (r) of greater than 0.995 acceptability. Calibration curves using the relative standard deviation (RSD) of RFs to determine linearity must meet the acceptability criteria specified within the method. For gas chromatography/mass spectrometer (GC/MS) methods, the average RF from the initial five-point calibration will be used to determine analyte concentrations. The continuing calibration (CCAL) will not be used to update the RFs from the initial five-point calibration. GC/MS methods also will meet all instrument performance and/or tuning criteria as specified by the methods.

Initial Calibration Verification

Initial calibration (ICAL) curves must be verified using a standard made from a source independent of the one used to make the ICAL standards. All target compounds must be included within the initial calibration verification (ICV), typically at a concentration around the midpoint of the calibration curve. Failure of the ICV requires corrective action as defined in Table 2.4.

Continuing Calibration and Verification

ICAL curves must be verified daily prior to sample analysis using a CCAL. All target compounds must be included within the CCAL, typically at a concentration around the midpoint of the calibration curve. CCALs are required at the beginning and end of each analytical sequence and after every 10 samples analyzed (or as specified in each analytical method). Failure of the CCAL requires corrective action as defined in Table 2.4.

Standard Reference Materials

Standard materials used in calibration and to prepare samples will be traceable to National Institute of Standards and Technology (NIST), EPA, American Association of Laboratory Accreditation (A2LA), or other equivalent approved source, if available. The standard materials will be current, in accordance with the following expiration policy: The expiration dates for ampulated solutions will not exceed the manufacturer's expiration date or 1 year from the date of receipt, whichever occurs first. Expiration dates for laboratory-prepared stock and diluted standards will be no later than the expiration date of the stock solution or material, or the date calculated from the holding time allowed by the applicable analytical method, whichever occurs first. The laboratory will label standard and QC materials with expiration dates.

TABLE 2.4
SUMMARY OF CALIBRATION AND QC PROCEDURES
NIROP
FRIDLEY, MINNESOTA

Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Volatile Organics SW8260B	At least five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF τ 0.30 (>0.10 for bromoform and >0.01 for chloromethane and 1,1-dichloroethane); and RSD for all calibration analytes δ 30%	Correct problem then repeat initial calibration to obtain acceptable %RSD and RFs
	Daily BFB Tune	Performed at the beginning of each 12 hour period during which sample analyses are performed	BFB Tune must meet the spectrum criteria defined in Appendix C, Table 1 of En Chem SOP – VOA-5	Modify the tune file again; if the BFB or the autotune fails repeatedly, source cleaning may be necessary
	Continuing calibration check standard	Performed at the beginning of each 12 hour period during which sample analyses are performed	Compare the daily response factors with the average response factors from the initial calibration. SPCCs average RF τ 0.30 (>0.10 for bromoform and >0.01 for chloromethane and 1,1-dichloroethane). The percent difference for any CCC compound may not be greater than 20%. In addition, the RRT for each analyte must compare within ± 0.06 RRT units.	Correct problem then repeat continuing calibration. Repeated failure to meet acceptance criteria is a sign that the initial calibration is not valid and should be repeated.

TABLE 2.4 (Continued)
SUMMARY OF CALIBRATION AND QC PROCEDURES
NIROP
FRIDLEY, MINNESOTA

Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
	IS	Every sample, spiked sample, standard, and method blank	Retention time ρ30 seconds: EICP area within -50% to +100% of last calibration verification (12 hours) for each	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
	Method blank	Performed at the beginning of each 12 hour period during which sample analyses are performed	1. Non common solvent may be present above 5 times the EQL 2. Target compound other than common solvents should not be greater than the reporting limit 3. Non-target compound peaks with areas greater than 10% of the area of the nearest IS, other than surrogates, must not be present in the chromatogram (if TICs are requested)	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes	Performed at the beginning of each 12 hour period during which sample analyses are performed	QC acceptance criteria based on CLP OLM3.0	Correct problem then reprep and analyze the LCS and all samples in the affected analytical batch

TABLE 2.4 (Continued)
SUMMARY OF CALIBRATION AND QC PROCEDURES
NIROP
FRIDLEY, MINNESOTA

Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria	Correct problem then reextract and analyze sample
	MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria	Re-extract and re-analyze the MS and MSD sample within holding time
	MDL study	Once per year	Detection limits established shall meet QAPP-established criteria	Re-establish MDL
Methane/ Ethane/ Ethene (SW5021)	Initial calibration verification	Minimum of 5 levels; lowest level near but above MDL.	Correlation co-efficient > 0.995	Correct problem then repeat initial calibration
	Continuing calibration verification	Mid-level calibration standards run every 10 samples and at the beginning and end of each 12 hour time clock.	Acceptable if recovery is 80-120%	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification
	Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.	No analytes detected > PQLs,	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank

TABLE 2.4 (Continued)
SUMMARY OF CALIBRATION AND QC PROCEDURES
NIROP
FRIDLEY, MINNESOTA

Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
	Laboratory control spike and duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent. Fortify with all target compounds.	Acceptable if recovery is 70-130% and RPD is < 20%	Correct problem then reprep and analyze the LCS and all samples in the affected analytical batch
	Internal standards	Not applicable	Not applicable	Not applicable
	Surrogate standards	Not applicable	Not applicable	Not applicable
	MS/MSD	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent. Fortify with all target compounds.	Acceptable if recovery is 70-130% and RPD is < 20%	Re-extract and analyze the MS and MSD sample within holding time

TABLE 2.4 (Continued)
SUMMARY OF CALIBRATION AND QC PROCEDURES
NIROP
FRIDLEY, MINNESOTA

Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Nitrate/ Nitrite E300.1	Six point initial calibration	Initial calibration prior to sample analysis	Correlation coefficient > 0.995 for linear regression	Correct problem then repeat initial calibration
	Initial calibration verification (ICV)	Immediately after calibration	Analyte within ρ10% of true value	Correct problem then repeat initial calibration
	APG Check standard	For each anion analyzed weekly	Analyte within ρ10% of true value	The separation column will be replaced or taken out of service until it can be cleaned. Record results in IC Daily Log
	Initial calibration blank (ICB)	ICB analyzed after ICV	Absolute value must be ≤ EQL	Recalibrate if ICB fails
	Continuing calibration verification (CCV)	After every 10 samples	Analyte within ρ10% of true value	Correct problem then repeat calibration and reanalyze all samples between CCV and CCB
	Continuing calibration blank (CCB)	After every CCV	Absolute value must be ≤ EQL	Correct problem then repeat calibration and reanalyze all samples between last CCB and CCV

TABLE 2.4 (Continued)
SUMMARY OF CALIBRATION AND QC PROCEDURES
NIROP
FRIDLEY, MINNESOTA

Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
	MS/MSD	Frequency of 5% of similar matrix	% Recovery 90-110 %RPD 4% max	If both spike recoveries are outside, the corresponding parent sample is to be diluted and MS/MSD performed. If only MS or MSD is out of control for accuracy, then the corresponding parent sample is flagged. If the RPD is out of control, the reported sample result is qualified with * flag.
	Method blank	Frequency of 5%	No analytes detected > EQL.	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank
	LCS for the analyte	Frequency of 5%	% Recovery 90-110	Correct problem then reprep and analyze the LCS and all samples in the affected analytical batch

TABLE 2.4 (Continued)
SUMMARY OF CALIBRATION AND QC PROCEDURES
NIROP
FRIDLEY, MINNESOTA

Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Total Organic Carbon E415.1	Calibration	Initial calibration prior to sample analysis	All analytes within ρ10% of true value	Correct problem then repeat initial calibration
(water) and SW846	Calibration check standard	Immediately after initial calibration	All analytes within ρ10% of true value	Correct problem then repeat initial calibration
Method 9060 (soil)	Initial calibration verification	Immediately after calibration check standard	All analytes within 10% of true value	Correct problem then repeat initial calibration
	Initial calibration blank (ICB)	ICB analyzed after ICV	Absolute value must be ≤EQL	Recalibrate if ICB fails
	Continuing calibration verification (CCV)	After every 10 samples	All analytes within 10% of true value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification

TABLE 2.4 (Continued)
SUMMARY OF CALIBRATION AND QC PROCEDURES
NIROP
FRIDLEY, MINNESOTA

Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
	Continuing calibration blank (CCB)	After every CCV	Absolute value must be ≤ EQL	Correct problem then repeat calibration and reanalyze all samples since last CCB
	Method blank	Frequency of 5%	No analytes detected > EQL	If MB is > EQL reprep and analyze method blank and all samples unless the sample concentrations are > 20X the method blank concentration. If the MB concentration is between MDL and EQL, all samples will be qualified with A flag unless the sample concentrations are > 20X the method blank concentration.
	LCS for all analytes	One LCS per analytical batch of 20 samples	% Recovery 86-130	Correct problem then reprep and analyze the LCS and all samples in the affected analytical batch
	MS/MSD	Frequency of 5% per matrix	% Recovery 35 – 155 %RPD 16% max	Flag results if either recovery or RPD is out.

TABLE 2.4 (Continued)
SUMMARY OF CALIBRATION AND QC PROCEDURES
NIROP
FRIDLEY, MINNESOTA

Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Hydrogen AM-20-GAX	4 point calibration curve with laboratory manufactured standards.	Daily	All analytes within 10% of expected value	Correct problem then repeat initial calibration
(modified SW8015)	Demonstrate ability to generate acceptable accuracy and precision using 1 replicate analyses of a QC check sample	Daily	QC acceptance criteria, lab-established	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria
	Method blank	One per analytical batch, or daily at minimum	No analytes detected > PQL,	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank

Note: All corrective actions associated with project work will be documented, and all records will be maintained by the laboratory. If discrepancies exist between this table and the laboratory SOPs, the laboratory SOPs take precedence.

2.5.2.3 Reporting Units

The prescribed reporting units for all analytical methods are as follow:

- ξ Soil samples - organics: micrograms per kilogram (μg/kg), dry-weight basis;
- ξ Soil samples - inorganics/metals: milligrams per kilogram (mg/kg), dry-weight basis;
- ξ Water samples - inorganics/metals: milligrams per liter (mg/L);
- ξ Water samples - organics: micrograms per liter (μg/L), and

All analytical results for soils (both non-detected and detected) will be reported on a dry-weight basis (i.e., corrected for moisture content). The moisture content for each soil sample will be reported. The equation for moisture content given for the Method SW3550 is as follows:

$$\frac{\text{Initial Weight - Dried Weight}}{\text{Initial Weight}} \times 100 = \%\ \text{moisture}$$

The result for the sample on a dry-weight basis is as follows:

$$\frac{\text{Result of analysis on wet-weight basis}}{100 - \%\ \text{Moisture}} = \text{Result of analysis on a dry-weight basis}$$

2.5.2.4 Detection and Quantitation Limits

This section describes the terms, definitions, and formulas that will be used for detection and quantitation limits.

Method Detection Limit

The MDL is the lowest concentration at which a specific analyte in a matrix can be measured and reported with 99-percent confidence that the analyte concentration is greater than zero. MDLs are experimentally determined and verified for each target analyte of the methods in the sampling program. The laboratory will determine MDLs for each analyte and matrix type prior to analysis of project samples. In addition, when multiple instruments are employed for the analysis of the same method, each individual instrument will maintain a current MDL study. MDLs are based on the results of seven matrix spikes at the estimated MDL, and are statistically calculated in accordance with the Title 40, Code of Federal Regulations Part 136 (40 CFR 136) Appendix B. The standard deviation of the seven replicates is determined and multiplied by 3.14 (i.e., the 99-percent confidence interval from the one-sided student t-test). If risk-based project objectives are developed, then where practicable, MDLs must be lower than the risk-based criteria determined for the project.

The MDLs to be used are intended to allow that both nondetected and detected target compound results will be usable to the fullest extent possible for the project. An MDL check sample (interference-free MS with all method target compounds) must be analyzed following the MDL study to determine if reasonable MDL concentrations have been

achieved. The MDL check sample should be at a concentration of approximately two times the MDL. If any target compound is not recovered, the MDL study must be repeated. In this case, the repeated MDL should be performed with a higher concentration, based on the analyst's judgment, of the target compounds that failed in the MDL check sample. MDLs must be determined annually at a minimum, and verified quarterly by analyzing an MDL check sample.

Sample Quantitation Limit

SQLs are defined as the laboratory reporting limit multiplied by the dilution factor (DF) required to analyze the sample, and corrected for moisture or sample size. These adjustments may be due to matrix effects or to the high concentrations of some analytes. For example, if an analyte is present at a concentration that is greater than the linear range of the analytical method, the sample must be diluted for accurate quantitation. The DF raises the reporting limit, which then becomes the SQL. Because the reported SQLs take into account sample characteristics and analytical adjustments, they are the most relevant quantitation limits for evaluating nondetected chemicals.

Project Reporting Limit

Target PRLs were selected using risk based concentrations (RBCs). PRLs are presented in Table 1.2. Laboratory reporting limits for each target compound are referred to as practical quantitation limits (PQLs). PQLs are laboratory-specific concentrations based on the MDL for each target compound. The PQLs will be equivalent to the concentration of the lowest calibration standard for organic analyses. The lowest calibration standard for all target compounds will be at a concentration between three and ten times the MDL. The PQL, and therefore the lowest calibration standard, must not exceed regulatory action levels (where practicable) for any target compound.

All target compound detections will be reported at or above the MDL for each analyte. All results above the MDL, but below the PQL, will be qualified in the data deliverable from the laboratory with a "J" flag for organic compounds or "B" flag for inorganics (metals). The "J or B" flags will denote the sample result as below the PQL, and as an estimated quantitative value. Laboratories must verify the PQLs by analyzing a standard at or below the PQL on a weekly basis at a minimum.

2.6 INSTRUMENT AND EQUIPMENT TESTING, INSPECTIONS, AND MAINTENANCE REQUIREMENTS

Equipment, instruments, tools, gauges, and other items requiring preventive maintenance will be serviced in accordance with the manufacturers' specified recommendations or written procedures developed by the operators.

2.6.1 Schedules

Manufacturers' procedures identify the schedule for servicing critical items to minimize downtime of the measurement system. It will be the responsibility of the individual operator assigned to a specific instrument to adhere to the instrument maintenance schedule and to promptly arrange any necessary service. Qualified

personnel will perform servicing of the equipment, instruments, tools, gauges, and other items.

The laboratory will establish logs to record maintenance and service procedures and schedules. All maintenance records will be documented and will be traceable to the specific equipment, instruments, tools, and gauges. All logbooks will be kept in an easily accessible location for ready reference by the laboratory analysts using the instrument. Entries include the date of service, type of problem encountered, corrective action taken, and initials and affiliation of the person providing the service. Records produced for laboratory instruments will be reviewed, maintained, and filed by the operators at the laboratories.

The instrument use logbook is monitored by the analysts to detect any degradation of instrument performance. Changes in response factors or sensitivity are used as indication of potential problems. These are brought to the attention of the laboratory supervisor and preventative maintenance or service is scheduled to minimize down time.

2.6.2 Spare Parts

A list of critical spare parts will be requested from manufacturers and identified by the operator. These spare parts will be stored for availability and use to reduce downtime due to equipment failure and repair.

2.7 INSTRUMENT CALIBRATION AND FREQUENCY

Calibration and QC procedures are presented in Table 2.4.

2.8 DATA MANAGEMENT

The data review process is performed in two phases. The initial phase, contract compliance screening (CCS), consists of inspecting the laboratory data deliverables to determine if the contract requirements were met. The second phase, data validation, includes a review of data results to assess data usability and application of data qualifiers to the analytical results based on adherence to method protocols and laboratory-specific QA/QC limits. Method SW8260B will undergo data validation.

2.8.1 Contract Compliance Screening

CCS is the review of sample data deliverables for completeness and compliance with project requirements. Completeness is evaluated by ensuring that all required data deliverables are received in a legible format with all required information. The CCS process also includes a review of the COC forms, case narratives, and PRLs. Sample resubmission requests, documentation of nonconformances with respect to data deliverable completeness, and corrective actions often are initiated during the CCS review.

2.8.2 Data Validation

Following completion of the CCS process, CH2M Hill will perform an EPA Level III-type validation on 10 percent of all analytical results. Because this project does not involve regulatory compliance monitoring, 100 percent validation of the data is not necessary. The validated data will be selected from the first round of analyses so that

potential problems can be identified early. The validation process includes a review of summary information to determine adherence to analytical holding times; results from analysis of field duplicates, method blanks, field blanks, surrogate spikes, MS/MSDs, LCSs, and sample temperatures during shipping and storage. The results of the CCS process are incorporated into the data validation process. Data qualifiers are applied to analytical results during the data validation process based on adherence to method protocols and laboratory-specific QA/QC limits.

The validation guidelines defined in Tables 2.5 and 2.6 are for use in validation, and were developed in accordance with the National Functional Guidelines for Organic Data Review (EPA, 1994b) and the National Functional Guidelines for Inorganic Data Review (EPA, 1994c) as modified for the specific analytical method. Expanded criteria for the validation guidelines were developed where professional judgment is recommended within the EPA guidelines. QC guidelines are those specified in the analytical method protocols.

Data qualified as rejected will be assessed as to their critical importance. If required the samples will be recollected and reanalyzed.

TABLE 2.5
FLAGGING CONVENTIONS FOR DATA EVALUATION AND VALIDATION OF ORGANIC METHODS
NIROP
FRIDLEY, MINNESOTA

Quality Control Check	Evaluation	Flag	Samples Affected
Holding Time	Holding time exceeded for extraction or analysis by > 2 times	J detects R non-detects	Sample
	Holding time exceeded for extraction or analyses by < 2 times	J detects UJ non-detects	Sample
Sample Preservation	Sample not preserved	J detects UJ non-detects	Sample
Temperature Blank	>8ºC	J detects UJ non-detects	All samples in same cooler
	>20ºC (Volatile Compounds)	R all results.	All samples in same cooler
Tune	Ion abundance criteria	J detects UJ non-detect results	All associated samples in analysis batch

TABLE 2.5 (Continued)
FLAGGING CONVENTIONS FOR DATA EVALUATION AND VALIDATION OF ORGANIC METHODS
NIROP
FRIDLEY, MINNESOTA

	Set critical ions as defined in SW846	R all detects R non-detects	All associated samples in analysis batch
ICAL	GC/MS: RRF <0.05	R non-detects J detects	Compound in all associated samples in analysis batch
	%RSD τ30% and all initial calibration RRF τ0.05	UJ non-detects J detects	Compound in all associated samples in analysis batch
	If %RSD >2X control criteria	R all detects R non-detects	Compound in all associated samples in analysis batch
CCAL	GC/MS: %D τ25% and RRFτ0.05	J detects UJ non-detects	Compound in all associated samples in analysis batch
	If %D is >2X control criteria	R all detects R non-detects	Compound in all associated samples in analysis batch
	RRF <0.05	J detects R non-detects	Compound in all associated samples in analysis batch
LCS and LCSD	LCS or LCSD single compound: %R < ½ LCL or 30% (whichever is lower)	R all detects R non-detects	Spiked compound only in all associated samples.
	%R >UCL but < UCL + ½ UCL	J detects No qualification for non-detects	Spiked compound only in all associated samples.
	% R ≥ ½ LCL or 30% (whichever is lower) but < LCL	J detects UJ non-detects	Spiked compound only in all associated samples.

TABLE 2.5 (Continued)
FLAGGING CONVENTIONS FOR DATA EVALUATION AND VALIDATION OF ORGANIC METHODS
NIROP
FRIDLEY, MINNESOTA

Quality Control Check	Evaluation	Flag	Samples Affected
	% R >UCL + ½ UCL	R all detects No qualification for non-detects	Spiked compound only in all associated samples.
	If ≥ 50% of all LCS or LCSD spiked compounds are out of control	R all detects R non-detects	All detected spike compounds in all samples
	RPD >control limit	J detects	All detected spike compounds in all samples
Method Blank	Multiply value by 5, common lab contaminants multiply by 10 (common lab contaminants: methylene chloride, acetone, 2-butanone, and phthalates)	U flag reported results < calculated value	All samples in extraction batch
Equipment Blank	Convert to soil units, if applicable, multiply by 5, common lab contaminants multiply by 10(common lab contaminants: methylene chloride, acetone, 2-butanone, and phthalates)	U flag reported results < calculated value	All associated samples
Trip Blank	Convert to soil units, if applicable, multiply by 5, common lab contaminants multiply by 10 (common lab contaminants: methylene chloride, acetone, 2-butanone, and phthalates)	U flag reported results < calculated value	All volatile samples shipped in the same cooler
MS/MSD	MS or MSD single compound: %R < ½ LCL or 10% (whichever is lower)	R all detects R non-detects	Affected compound in native sample MS/MSD
	%R >UCL but < UCL + ½ UCL	J detects No qualification for non-detects	Affected compound in native sample MS/MSD

TABLE 2.5 (Continued)
FLAGGING CONVENTIONS FOR DATA EVALUATION AND VALIDATION OF ORGANIC METHODS
NIROP
FRIDLEY, MINNESOTA

Quality Control Check	Evaluation	Flag	Samples Affected
	% R ≥ ½ LCL or 10% (whichever is lower) but < LCL	J detects UJ non-detects	Affected compound in native sample MS/MSD
	% R >UCL + ½ UCL	R all detects No qualification for non-detects	Affected compound in native sample MS/MSD
	If ≥ 50% of all MS or MSD spiked compounds are out of control:	R all detects R non-detects	All compounds in native sample
	When sample conc. is >4X spike conc.	No evaluation required	None
	RPD > control limit	J detects	Affected compound in native sample MS/MSD
Surrogates GC/MS VOCs	%R > UCL	J detects	All compounds in associated sample
	%R < LCL and τ 10%	J detects UJ non-detects	All compounds in associated sample
	%R < 10%	J detects R non-detects	All compounds in associated sample
Internal Standards (IS) (GC/MS)	RT change > UCL from daily CCAL	R all detects R non-detects	All associated compounds in sample
	IS extracted ion area counts < -50% to +100% of last CCAL	J detects UJ non-detects	All associated compounds in sample
	IS extracted ion area counts > +100% of last CCAL	J detects No qualification for non-detects	All associated compounds in sample
	IS extracted ion area counts < 10% of last CCAL	R all detects R non-detects	All associated compounds in sample

TABLE 2.5 (Continued)
FLAGGING CONVENTIONS FOR DATA EVALUATION AND VALIDATION OF ORGANIC METHODS
NIROP
FRIDLEY, MINNESOTA

Quality Control Check	Evaluation	Flag	Samples Affected
Retention Time Windows (RTW)	Analyte peak not within RTW	Report detects as non-detect, (professional judgment should be used prior to eliminating detections)	All affected compounds
Field Duplicates	RPD > 25% water , >35% for soil	Discuss impacts in data quality assessment report	Field duplicate pair

Acronyms:

%D - Percent difference.
%R - Percent recovery.
CCAL - Continuing calibration
GC/MS - Gas chromatograph/mass spectroscopy
HPLC - High performance liquid chromatography
ICAL - Initial Calibration
LCL - Lower control limit
LCS - Laboratory control sample
LCSD - Laboratory control sample duplicate
MS/MSD - Matrix spike/matrix spike duplicate
NFG - National Functional Guidelines
PCB - Polychlorinated biphenyl
RPD - Relative percent difference
RRF - Relative response factor
RT - Retention time
RSD - Relative standard deviation
SVOC - Semivolatile organic compounds
UCL - Upper control limit
VOCs - Volatile organic compounds

TABLE 2.6
FLAGGING CONVENTIONS FOR DATA EVALUATION AND VALIDATION OF INORGANIC METHODS
NIROP
FRIDLEY, MINNESOTA

Quality Control Check	Evaluation	Flag	Samples Affected
Holding Time	Holding time exceeded for digestion or analysis by < 2 times	J detects UJ non-detected results	Sample only
	exceeded by > 2 times	J detects R non-detects.	Sample only
Sample Preservation	Sample preservation requirements not met	J detects UJ non-detects for all methods except mercury R mercury non-detects	Sample only
Temperature Blank	>8ΘC	J detects UJ non-detects	Samples in same cooler
Initial (Multipoint) Calibration	Correlation coefficient of curve < 0.995 but > 0.990	J detects UJ non-detects	All associated samples in analysis batch
	Correlation coefficient of curve < 0.990	R detects R non-detects	All associated samples in analysis batch
Calibration Verification: ICV, CCV	%R between 111-125%	J detects No qualification for non-detects	All associated samples in analysis batch for ICV, Samples after failed CCV until next in control CCV
	%R > 125%	R detects No qualification for non-detects	All associated samples in analysis batch for ICV, Samples after failed CCV until next in control CCV

TABLE 2.6 (Continued)
FLAGGING CONVENTIONS FOR DATA EVALUATION AND VALIDATION OF INORGANIC METHODS
NIROP
FRIDLEY, MINNESOTA

Quality Control Check	Evaluation	Flag	Samples Affected
Calibration Verification: ICV, CCV (Cont'd)	%R between 75-89%	UJ non-detects J detects	All associated samples in analysis batch for ICV, Samples after failed CCV until next in control CCV
	%R < 75%	R detects R non-detects	All associated samples in analysis batch for ICV, Samples after failed CCV until next in control CCV
LCS/LCSD	LCS or LCSD single analyte: %R < ½ LCL or 30% (whichever is lower)	R all detects and non-detects	Spiked compound only in all associated samples.
	%R >UCL but < UCL + ½ UCL	J detects No qualification for non-detects	Spiked compound only in all associated samples.
	% R ≥ ½ LCL or 30% (whichever is lower) but < LCL	J detects UJ non-detects	Spiked compound only in all associated samples.
	% R >UCL + ½ UCL	R all detects No qualification for non-detects	Spiked compound only in all associated samples.
	If ≥ 50% of all LCS or LCSD spiked compounds are out of control:	R all detects R non-detects	All compounds in all associated samples
	RPD > control limit	J detects	Spiked compound only in all associated samples.
Blanks: MB, ICB, CCB	If the absolute value of the blank is >MDL, then multiply value by 5, convert to soil units if applicable	U flag reported results < calculated values	All samples in digestion batch (MB) All samples in analysis batch (ICB, CCB)

TABLE 2.6 (Continued)
FLAGGING CONVENTIONS FOR DATA EVALUATION AND VALIDATION OF INORGANIC METHODS
NIROP
FRIDLEY, MINNESOTA

Quality Control Check	Evaluation	Flag	Samples Affected
Equipment Blank	If the absolute value of the blank is >MDL, then multiply value by 5, convert to soil units if applicable	U flag reported results < calculated values	All associated samples
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	MS or MSD single analyte:		
	%R < ½ LCL or 30% (whichever is lower)	J detects R non-detects	Affected compound in native sample
	%R >UCL but < UCL + ½ UCL	J detects No qualification for non-detects	Affected compound in native sample
	% R ≥ ½ LCL or 30% (whichever is lower) but < LCL	J detects UJ non-detects	Affected compound in native sample
	% R >UCL + ½ UCL	R all detects No qualification for non-detects	Affected compound in native sample
	If ≥ 50% of all MS or MSD spiked compounds are out of control:	R all detects R non-detects	All compounds in native sample
	When sample conc. is >4X spike conc.	No evaluation required	None
	RPD > control limit	J-detects No qualification for non-detects	Affected compound in native sample
Field duplicates	RPD > 25% water RPD > 35%soil	Discuss in data quality assessment report	Field duplicate pair

Acronyms:

%R - Percent recovery.
CCB - Continuing calibration blank.
CCV - Continuing calibration verification
ICB - Initial calibration blank
MDL - Method detection limit
MS/MSD - Matrix spike/matrix spike duplicate
r - Correlation coefficient
RPD - Relative percent difference.

ICV - Initial calibration verification
LCL - Lower control limit
LCS - Laboratory control sample
LCSD - Laboratory control sample duplicate
MB - Method blank.
UCL - Upper control limit.

2.8.3 Data Validation Qualifiers

The following definitions provide explanations of the EPA (1994b and 1994c) qualifiers to be assigned to analytical results during data validation, in accordance with Tables 2.5 and 2.6. The data qualifiers described are applied to both inorganic and organic results.

U - The analyte was analyzed for and is not present above the reported SQL.

J - The analyte was analyzed for and was positively identified, but the associated numerical value may not be consistent with the amount actually present in the environmental sample. The data should be considered as a basis for decision-making and are usable for many purposes.

R - The data are rejected as unusable for all purposes. The analyte was analyzed for, but the presence or absence of the analyte was not verified. Resampling and reanalysis are necessary to confirm the presence or absence of the analyte.

UJ - The analyte was not present above the reported SQL. The associated numerical value may not accurately or precisely represent the concentration necessary to detect the analyte in the sample.

SECTION 3.0

ASSESSMENT AND OVERSIGHT

3.1 ASSESSMENT AND RESPONSE ACTIONS

3.1.1 Planned Assessments

This section describes participation in external and internal systems audits for contractors and laboratories. There are no external audits scheduled for this project.

3.1.1.1 System Audits

System audits review laboratory operations and the resulting documentation. An onsite audit ensures that the laboratory has all the personnel, equipment, and internal SOPs needed for performance of contract requirements in place and operating. The system audits ensure that proper analysis documentation procedures are followed, that routine laboratory QC samples are analyzed, and that any nonconformances are identified and resolved.

Internal Audits

The laboratory must conduct internal system audits on a periodic basis. The Laboratory QA Officer will document the results of these audits, and the laboratory will provide the contractor with the results of these internal audits.

External Audits – Fixed Laboratory Technical Systems Audit

The contractor Project QA Officer or designee may conduct an external system audit of the laboratory during the performance project samples. This audit would evaluate the capabilities and performance of laboratory personnel, items, and activities. It also documents the measurement systems and identifies and corrects any deficiencies. The contractor QA Manager acts on audit results by documenting deficiencies and informing the contractor PM of the need for corrective action. The contractor PM may suspend operations until problems are resolved. If conditions adverse to quality are detected, or if the contractor PM requests additional audits, additional unscheduled audits may be performed.

In addition to the contractor audit of the laboratory, various state and/or federal agencies may conduct an audit prior to the commencement of the project, and may conduct additional audits as deemed necessary. The frequency and schedule of any such audits will be established by the auditing agency and coordinated directly with the laboratory.

3.1.1.2 Performance Evaluation Sample Tracking and Analysis Audits

Laboratory performance audits may be conducted to determine the accuracy and implementation of the QAPP by the contractor Project QA Officer or designee prior to initiation of field sampling. Unplanned audits may be implemented if requested by the

contractor PM. In addition to in-house performance audits, the laboratory may also participate in interlaboratory performance evaluation studies for different state or federal agencies. The contractor Project QA Officer will act to correct any laboratory performance problems.

3.1.1.3 Field Sampling Technical System Audits and Field Analytical Technical System Audits

The mobilization stage may be audited before work begins to ensure that all procedures, training, and materials are ready to support the QAPP. Field activities may be audited during operation in order to assure compliance with the QAPP. Additional audits may be required depending on the results of these audits.

3.1.2 Assessment Findings and Corrective Action Responses

The following procedures have been established to ensure that conditions adverse to data quality are promptly investigated, evaluated, and corrected. Adverse conditions may include malfunctions, deficiencies, deviations, and errors. When a significant condition adverse to data quality is noted at the laboratory or in the field, the cause of the condition will be determined and corrective action will be taken to prevent repetition. Condition identification, cause, reference documents, and corrective action planned will be documented and reported to the contractor project QA officer by the laboratory QA officer. Following implementation of corrective action, the laboratory QA officer will report the actions taken and their results to the contractor Project Manager and QA Officer. A record of the action taken and results will be attached to the data report package. If samples are reanalyzed, the assessment procedures will be repeated, and the control limits will be reevaluated to ascertain if corrective actions have been successful.

Implementation of corrective action is verified by documented follow-up action. All project personnel have the responsibility, as part of the normal work duties, to identify, report, and solicit approval of corrective actions for conditions adverse to data quality.

Corrective actions will be initiated in the following instances:

- When predetermined acceptance criteria are not attained (objectives for precision, accuracy, and completeness);
- When the prescribed procedure or any data compiled are faulty;
- When equipment or instrumentation is determined to be faulty;
- When the traceability of samples, standards, or analysis results is questionable;
- When QA requirements have been violated;
- When designated approvals have been circumvented;
- As a result of systems or performance audits;
- As a result of regular management assessments;

- As a result of intralaboratory or interlaboratory comparison studies; and
- At any other instance of conditions significantly adverse to quality.

Laboratory project management and staff, such as QA auditors, document and sample control personnel, and laboratory groups, will monitor work performance in the normal course of daily responsibilities.

The Laboratory QA Officer or designated alternate will audit work at the laboratory. Items, activities, or documents ascertained to be compliant with QA requirements will be documented, and corrective actions will be mandated in the audit report. The contractor Project QA Officer and Laboratory QA Officer will log, maintain, and control the audit findings.

The contractor Project QA Officer and Laboratory QA Officer are responsible for documenting all out-of-control events and nonconformances with QA protocols. The QC checks, their frequency, acceptance criteria, and corrective actions for out-of-control data are summarized in Table 2.4 for each analytical method. A nonconformance report will summarize each nonconformance condition. No specific format for the nonconformance report is required. The report should state the problem and address the laboratory's process for corrective action. The laboratory will notify the contractor Project Manager or QA Officer of any laboratory QA/QC nonconformance upon discovery. Copies of all field change requests and corrective action forms will be maintained in the project files. The contractor may initiate a stop-work order if corrective actions are insufficient.

3.2 REPORTS TO MANAGEMENT

At monthly intervals beginning with the initiation of sampling activities, the laboratory will submit to the contractor's project task manager a QA report that documents laboratory-related QA/QC issues. These reports will include discussions of any conditions adverse or potentially adverse to quality, such as:

- Responses to the findings of any internal or external systems or performance laboratory audits;
- Any laboratory or sample conditions that necessitate a departure from the methods or procedures specified in this QAPP;
- Any missed holding times or problems with laboratory QC acceptance criteria; and
- The associated corrective actions taken.

Submittal of QA reports will not preclude earlier contractor notification of such problems when timely notice can reduce the loss or potential loss of quality, time, effort, or expense. Appropriate steps will be taken to correct any QA/QC concerns as they are identified. The QA reports and a summary of the laboratory QA/QC program and results will be included in the final project report.

SECTION 4.0

DATA VERIFICATION, VALIDATION AND USABILITY

4.1 DATA REVIEW, VALIDATION AND VERIFICATION REQUIREMENTSAND METHODS

4.1.1 Contractor Data Validation

Contractor data validation procedures are described in Section 2.8.1.

4.1.2 Laboratory Data Review

The following sections describe laboratory data review and reporting.

4.1.2.1 Review Procedures for Definitive Data

The laboratory review of definitive data is a four-step process involving an evaluation by the analyst, a peer review, an administrative review, and a QA review. A checklist to document each of the review processes will be required and must be included as part of the final data deliverable to the contractor. All steps are described below.

The analyst will review 100 percent of all definitive data prior to reporting. The establishment of detection and control limits will be verified. Any control limits outside of the acceptable ranges specified in the analytical methods will be identified. Any trends or problems with the data will be evaluated. The absence of records supporting the establishment of control criteria or detection limits will be noted. Analytical batch QC, calibration check samples, ICALs, CCVs, corrective action reports, the results of reanalysis, sample holding times, and sample preservations will be evaluated.

Samples associated with out-of-control QC data will be identified in the data package case narrative, and an assessment of the utility of such analytical results will be made. The check of laboratory data completeness must be documented and will ensure that:

- ξ All samples and analyses specified in the COC have been processed;
- ξ Complete records exist for each analysis and the associated QC samples; and
- ξ Procedures specified in this QAPP have been implemented.

An analyst other than the original data processor will be responsible for performing a peer review of all steps of the data processing. A minimum of 25 percent of all data will be reviewed. All input parameters, calibrations, and transcriptions will be checked. All manually input, computer-processed data will be checked. Data review checklists will be used to document second level/peer review.

QC sample results (LCSs, MS/MSDs, surrogates, and ICAL and CCAL standards) are compared against stated criteria for accuracy and precision. QC data must meet acceptance levels prior to processing the analytical data. If QC standards are not met, the

cause will be determined. If the cause can be corrected without affecting the integrity of the analytical data, processing of the data will proceed. If the resolution jeopardizes the integrity of the data, reanalysis will occur.

An administrative review will be performed by the laboratory project manager on each data deliverable package. The review will ensure that all requirements of the laboratory and the data deliverable have been met and are complete.

A review of at least 10 percent of all data deliverable packages from a laboratory QA officer must take place prior to the administrative review and final release of the data deliverable to the contractor. The data packages will be randomly selected for review.

Decisions to repeat sample collection and analyses may be made by the contractor project manager based on the extent of the deficiencies and their importance in the overall context of the project.

4.2.1.2 Laboratory Data Reporting Flags

The laboratory must use the following qualifiers when reporting sample results.

Laboratory Organic Data Reporting Qualifiers

The laboratory must use the following qualifiers when reporting results of organic analyses:

Value- If the result is a value greater than or equal to the PQL, the value is reported.

U- Indicates the compound was analyzed for but not detected. The number is the project reporting limit (e.g., the nondetect limit) for the sample.

J- Indicates an estimated value. This flag is used to estimate a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicate identification criteria, but the result is less than the specified detection limit. This flag will also be used to identify values falling between the MDL and the PQL.

C- Applies to PCB parameters when the identification has been confirmed by GC/MS.

B- Used when the analyte is found in the blank, as well as a sample. It indicates possible/probable blank contamination and warns data user to take appropriate action.

E- Identifies compounds whose concentrations exceed the calibration range of the instruments for specific analysis.

D- Identifies all compounds analyzed at a secondary dilution.

A- Indicates that a tentatively identified compound (TIC) is a suspected aldol-condensation product.

X- Any other specific flags and footnotes that may be required to properly define the results.

RE- Analysis performed on a re-extracted sample.

Laboratory Inorganic Data Reporting Qualifiers

The laboratory must use the following qualifiers when reporting results of inorganic analyses.
Concentration Qualifiers:

B- The reported value was obtained from a reading that was less than the PQL, but greater than or equal to the MDL or instrument detection limit (IDL).

U- The analyte was analyzed for but not detected. The number is the project reporting limit (e.g., the nondetect limit) for the sample.

Q- Qualifier for specified entries:

E- The reported value is estimated due to the presence of interference(s).

M- Duplicate injection precision not met.

N- Spike sample recovery not within control limits.

S- The reported value was determined by the method of standard additions (MSA).

W- Post-digestion spike for GFAA analysis is out of control limits (85-115%), while sample absorbance is less than 50% of spike absorbance.

***-** Duplicate analysis not within control limits.

+- Correlation coefficient for the MSA is less than 0.995.

D- Spike level under IDL with dilution.

(The use of "S", "W", or "+" is mutually exclusive. No combination of these qualifiers should appear in the same field for an analyte.)

Method Qualifiers:

P- ICP.

A- Flame atomic absorption (FAA).

F- Furnace AA.

CV- Manual cold-vapor AA.

AV- Automated cold-vapor AA.

AS- Semi-automated spectrophotometric.

C- Manual spectrophotometric.

T- Titrametric.

NR- Analyte not required to be analyzed.

4.1.3 Review of Field Records

All field records will be evaluated for the following QC parameters:

4.1.3.1 Completeness of field records.

The check of field record completeness will ensure that all requirements for field activities in the site-specific work plan have been fulfilled, that complete records exist for each field activity, and that the procedures specified in the QAPP (or approved as field change requests) were implemented. Field documentation will ensure sample integrity and provide sufficient technical information to recreate each field event. The results of the completeness check will be documented, and environmental data affected by incomplete records will be identified in the technical report. The Project Manager will be responsible for ensuring that all scoped field analyses have been performed during sampling activities at each site. The completeness check will be performed on a daily basis.

4.1.3.2 Identification of valid samples.

The identification of valid samples involves interpretation and evaluation of the field records to detect problems affecting the representativeness of environmental samples. For example, field records can indicate if a well is properly constructed or if unanticipated environmental conditions were encountered during construction. Lithologic and geophysical logs may be consulted to determine if a well was screened only in the water-bearing zone of concern. Records also should note sample properties such as clarity, color, and odor. Photographs may show the presence or absence of obvious sources of potential contamination, such as combustion engines in operation near a well during sampling. Judgments of sample validity will be documented in the technical report, and environmental data associated with poor or incorrect fieldwork will be identified.

4.1.3.3 Correlation of data.

The results of field tests obtained from similar areas will be correlated. For example, photo-ionization detector (PID) readings and VOC analysis results may be correlated. The findings of these correlations will be documented, and the significance of anomalous data will be discussed in the technical report.

4.1.3.4 Identification of anomalous field test data.

Anomalous field data will be identified and explained to the extent possible. For example, a water temperature for one well that is significantly higher than any other well temperature in the same aquifer will be explained in the technical report.

4.1.3.5 Accuracy and precision of field data and measurements.

The assessment of the quality of field measurements will be based on instrument calibration records and a review of any field corrective actions. Accuracy will be based on check sample results (within control limits) and the frequency of calibration. Precision will be based on replicate measurements during analysis. The accuracy and precision of field measurements will be discussed. Field record review is an ongoing process. Field team leaders will be responsible for ensuring that proper recording is performed during sampling activities at each site.

4.2 USABILITY AND RECONCILIATION WITH DATA QUALITY OBJECTIVES

Data from QC samples will be assessed by the contractor using the procedures and criteria presented earlier in this section. In addition, the contractor will assess the usability of analytical data. Any limitations on data use will be expressed quantitatively to the extent practicable. This data usability review will include a review of the analytical methods, quantitation limits, and other factors important in determining the PARCC parameters. The outcome of this data review will be a data set appropriate to support project-specific DQOs. A data quality assessment (DQA) will be written and submitted by the contractor, if required, summarizing the findings of the review, and providing an assessment of overall data quality and usability.

Screening data will constitute all analytical method results from analyses performed in a field laboratory environment. The contractor will determine if the DQOs for field data have been met, and also will calculate the PC for field data results.

At a minimum, the review of screening data will focus on the following topics:

- ξ Holding times;
- ξ Method blanks;
- ξ Field instrumentation calibration and detection limits;
- ξ Completeness of data.

Field data will be validated using four different procedures, as described below:

- ξ Routine checks (e.g., looking for errors in identification codes) will be made during the processing of data.
- ξ Internal consistency of a data set will be evaluated. This step will involve plotting the data and testing for outliers.

- ξ Checks for consistency of the data set over time will be performed. This can be accomplished by comparing data sets against gross upper limits obtained from historical data sets, or by testing for historical consistency. Anomalous data will be identified.

- ξ Checks may be made for consistency with parallel data sets. An example of such a check would be comparing data from the same region of the aquifer or volume of soil.

The Project Manager (PM) will be responsible for field data assessment at the end of the project. A discussion of the field data review will be included in the technical report.

SECTION 5

REFERENCES

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EPA. 1995. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW846, (3rd Edition, Update III). United States Environmental Protection Agency (EPA).

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ATTACHMENT 1

ANALYTICAL LABORATORY ELECTRONIC DELIVERABLE REQUIREMENTS

The electronic data deliverable (EDD) file from the laboratory will be a comma-delimited ASCII (CDA) file in the format listed below. There will be one file per hard copy report and the filename of the EDD file will be in the format REPORTID.txt or REPORTID.csv, where REPORTID is the hard copy report identifier of sample delivery group.

The first row of the EDD will contain the 47 field name values as listed in the EDD Specification Table

The EDD Specification Table lists the attributes of the columns for each row of the CDA file. The fields should be reported in the order indicated.

The Data Type column describes the value in the field as either text (alphanumeric), number (numeric only), date (format: mm/dd/yyyy), or time (24-hour format hh:mm). If the field is conditional or optional and there is no value to be reported, report a null (i.e., no) value. For a text field, do not report a zero-length string (i.e., "").

The Data Length column contains the maximum length of a text value for the particular data field.

The Rqmt column contains a code indicating whether the value is required (R) for all rows, optional (O) for all rows, or conditional (C) and depends on the type of result reported.

EDD Specification Table					
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**Description and Comments**
1	VersionCode	text	15	R	Code identifying the version of the EDD deliverable.
2	LabName	text	10	R	Identification code for the laboratory performing the work. This value is used to distinguish among different facilities.
3	SDG	text	8	R	Sample delivery group designation. Always populated for all samples, including QC.
4	FieldID	text	13	R	Client sample ID as appears on COC with optional lab-assigned suffixes and/or prefixes to make it unique. If the sample identifier on the COC and the prefix/suffix is greater than 13 characters, abbreviate the value but make it unique. For laboratory QC samples (i.e., method blanks, lab control samples), use a unique lab sample identifier.
5	NativeID	text	13	R	Client sample ID, exactly as on the COC. No prefix or suffix allowed. Used to identify the native sample from which other samples are derived (e.g., QAQCType = "LR", "MS", or "SD"). For laboratory QC samples (i.e., method blanks, lab control samples), use a unique lab sample identifier. For lab blank spike (and blank spike duplicate) samples, use the FieldID value that was assigned to the associated method blank.

EDD Specification Table					
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**Description and Comments**
6	QAQCType	text	2	R	This is the code for the sample type. Any field sample that is not used as lab QC and is not otherwise marked on the COC should have the designation of "N" (normal field sample). No suffix allowed (i.e., do not add numbers as suffixes to the QAQCType values as is called for in the ERPIMS guidelines). Note that if all analyses for a given sample are diluted, then the first dilution should be designated as the normal sample. If more dilutions are required, then the next dilution should be designated as the first true dilution with a QAQCType value of "LR" and a LRType value of "DL" (see LRType, below).
7	LRType	text	3	C	This is the code for laboratory replicate sample type. Values are: blank (if QAQCType value is not "LR"), "DL" (dilution), "RE" (re-analysis), "D" (inorganic duplicate), "CF" (confirmation). For multiple dilutions or re-analyses of the same sample, append the replicate number after the LRType value (i.e., "RE", "RE2", "RE3", etc.).

EDD Specification Table					
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**Description and Comments**
8	Matrix	text	5	R	Sample matrix code. Valid values are as follows: "AIR", "WATER", "SOIL", unless otherwise provided by the project data manager and marked on the COC. The use of "liquid", "solid", etc. for lab QC is not allowed.
9	LabSampleID	text	20	R	Laboratory sample ID. Prefix or suffix is allowed. This is where dilutions or re-extractions are noted. Ex: "D97-11111RE" is acceptable.
10	AnalysisMethod	text	20	R	Analysis method code. This is the identifier of the analytical method that was performed on the sample. Example: SW8260A. Generic names such as "EPA" should not be used.
11	ExtractionMethod	text	20	R	Preparation method code. A value in this field is required. If the preparation is described in the method, use "METHOD". If there is no separate preparation required, use "NONE". Note that Total and Dissolved metal analyses are differentiated by the value in this column. Note that Total, TCLP, and SPLP analyses are now differentiated by the value in the LeachMethod column (see below).
12	SampleDate	date		C	Date of sample collection. Value is required for all samples sent to the laboratory and samples derived from those samples. Format: mm/dd/yyyy
13	SampleTime	time		C	Time of sample collection. Value is required for all samples sent to the laboratory and samples derived from those samples. 24-hour format: hh:mm

EDD Specification Table					
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**Description and Comments**
14	ReceiveDate	date		C	Date of sample receipt in the lab. Value is required for all samples sent to the laboratory and samples derived from those samples. Format: mm/dd/yyyy
15	ExtractDate	date		C	Date of sample preparation (extraction or digestion). Value is required if the ExtractionMethod field value is other than "NONE". Format: mm/dd/yyyy
16	ExtractTime	time		C	Time of sample preparation. Value is required if the ExtractionMethod field value is other than "NONE". 24-hour format: hh:mm
17	AnalysisDate	date		R	Date of sample analysis. Value is required for all records. Format: mm/dd/yyyy
18	AnalysisTime	time		R	Time of sample analysis. Value is required for all records. 24-hour format: hh:mm
19	PercentSolids	number		R	Percent solids within the sample. Should be zero for water samples.
20	LabLotCtlNum	text	10	C	Identifier of an autonomous group of environmental samples and associated QC samples **prepared** together. For example, its value can be a digestion or extraction batch ID. If there is no separate extraction or preparation performed, leave this field blank.
21	CAS	text	20	C	CAS number of analyte, if available.

EDD Specification Table					
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**Description and Comments**
22	ParamID	text	12	R	Parameter identifier code for the parameter listed in the Analyte field.
23	Analyte	text	60	R	Name of analyte, chemical name.
24	Result	text	10	R	Result of the analysis. Surrogate analytes will be reported in units of percent. All others will be reported in sample concentration units. If undetected, report the adjusted MDL or adjusted RL, depending on the project. (Reported as a text field to preserve significant figures.)
25	ExpectedValue	number		C	"100" for surrogates; "0" (zero) for blanks; spike level plus parent result for LCS, and MS/MSD; parent value for lab duplicate; etc.
26	Units	text	10	R	Units of measure used in the analysis. Report "PERCENT" for surrogate analytes and concentration units for all others.
27	Dilution	number		R	Total dilution reported in the analysis. Default value should be 1 (one). This value should reflect changes to sample preparation amounts as defined by the method (e.g., less sample used for standard VOC analysis).
28	MDL	number		C	Minimum detection limit adjusted for preparation and dilution. Note that this value may be the method detection limit or the instrument detection limit, depending on the method and the project requirements. This value is **not** adjusted for percent moisture.
29	RL	number		C	Reporting limit adjusted for preparation and dilution. Value is **not** adjusted for percent moisture. Equivalent to PQL.

EDD Specification Table					
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**Description and Comments**
30	LabQualifier	text	6	R	Lab qualifier for the results, as reported on the hard copy. Use "=" as first (or only) qualifier value for detected results.
31	Surrogate	text	1	R	Is the chemical a surrogate? Report "Y" for yes or "N" for no.
32	Comments	text	240	O	Comment field
33	ParValUncert	text	16	C	Radiological parameter value uncertainty.
34	Recovery	number		C	Percent recovery for MS, SD, LCS, and surrogate compounds.
35	LowerControlLimit	number		C	Lower control limit value for spiked compounds, expressed in units of Percent. A value in this field is required if there is a value in the Recovery field (Field No. 34).
36	UpperControlLimit	number		C	Upper control limit value for spiked compounds, expressed in units of Percent. A value in this field is required if there is a value in the Recovery field (Field No. 34).
37	Basis	text	1	R	Weight basis for soil (or solid) sample analysis. Use "D" for dry-weight basis, "W" for wet-weight basis, or "X" if not applicable.
38	ConcQual	text	1	R	Concentration qualifier. Use "=" for detects, "J" for estimated value (value between detection limit and reporting limit), "U" for undetected result, or "E" for exceeded result.

EDD Specification Table					
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**Description and Comments**
39	MDLAdjusted	number		C	Minimum detection limit adjusted for preparation, dilution **and percent moisture**. See the description of the MDL field (Field No. 28) for an explanation of the contents of this field.
40	RLAdjusted	number		C	Reporting limit adjusted for preparation, dilution **and percent moisture**. Equivalent to PQL
41	SampleDescription	text	20	C	Full sample identifier value as it appears on the COC. In some cases, this may be the name of the sampling location instead of the sample. Required for all samples that are either collected in the field and specified on the COC, or derived from samples that are collected in the field and specified on the COC.
42	LeachMethod	text	20	R	Analytical method used for leaching the sample. This applies to TCLP, SPLP, or other leaching or pre-extraction leaching procedures. Use "NONE" if the sample was not leached.
43	LeachDate	date		C	Date that the leaching method was performed (start date for multi-date leaching procedures). Value is required if the LeachMethod field value is other then "NONE". Format: mm/dd/yyyy.
44	LeachTime	time		C	Time that the leaching procedure started. Value is required if the LeachMethod field value is other then "NONE". 24-hour format: hh:mm.
45	LeachLot	text	20	C	Identifier of an autonomous group of environmental samples and associated QC samples **leached** at the same time. If the sample was not leached, leave this field blank.

EDD Specification Table					
Field Number	**Field Name**	**Data Type**	**Data Length**	**Rqmt**	**Description and Comments**
46	AnalysisLot	text	20	R	Identifier of an autonomous group of environmental samples and associated QC samples **analyzed** together. A value in this field is mandatory (i.e., it should not be blank).
47	CalRefID	text	20	C	Identifier of a group of environmental and QC samples linked by a common set of calibration records. All results with the same CalRefID value will have had the same initial calibration run.

Each row is uniquely identified by the values in the following fields:

- FieldID
- AnalysisMethod
- ExtractionMethod
- LeachMethod
- ParamID

If an analytical sample must be diluted or reanalyzed and reported in addition to the original analytical sample, the diluted or reanalyzed sample should have a FieldID value that is different that that of the original sample. This can be accomplished through the addition of a suffix to the original FieldID that establishes a new and unique FieldID for the associated records.

Example Valid Values

The project data manager will provide the laboratory with a list of valid values that the laboratory will use in constructing the EDD. Listed below are some example valid values.

Field Name	Valid Value	Meaning
VersionCode	4.00AFCEE3	Format 4.00, AFCEE data values. LabQualifier field contains the laboratory qualifier values defined in the AFCEE QAPP, version 3.0.
VersionCode	4.00EPACLP	Format 4.00, EPA data values. LabQualifier field contains the standard EPA CLP lab qualifiers.
QAQCType	N	Normal, environmental sample
QAQCType	LB	Laboratory method blank
QAQCType	MS	Laboratory matrix spike sample
QAQCType	SD	Laboratory matrix spike duplicate
QAQCType	LR	Laboratory replicate (dilution, re-analysis, duplicate)
QAQCType	BS	Laboratory method blank spike
QAQCType	BD	Laboratory method blank spike duplicate
LRType	DL	First dilution sample
LRType	DL2	Second dilution sample
LRType	DL3	Third dilution sample
LRType	RE	First re-analysis/re-extraction sample

Field Name	Valid Value	Meaning
LRType	RE2	Second re-analysis/re-extraction sample
LRType	RE3	Third re-analysis/re-extraction sample
LRType	D	Inorganic duplicate sample
LRType	CF	First confirmation analysis sample
LRType	CF2	Second confirmation analysis sample
LRType	CF3	Third confirmation analysis sample
AnalysisMethod	SW8260A	Volatiles by method 8260A in EPA SW846.
AnalysisMethod	SW8270	Semivolatiles by method 8270 in EPA SW846.
AnalysisMethod	SW6010	ICP metals by method 6010 in EPA SW846.
AnalysisMethod	SW7060	GFAA Arsenic by method 7060 in EPA SW846.
ExtractionMethod	FLDFLT	Field filtration for dissolved metals analysis
ExtractionMethod	C3050	CLP-modified SW3050 acid digestion for metals analysis in soil samples.
ExtractionMethod	SW1311	TCLP extraction
ExtractionMethod	DISWAT	Distilled water extraction for analytes in soil samples.
ExtractionMethod	SW3510	Separatory funnel extraction

Field Name	Valid Value	Meaning
ExtractionMethod	SW3540	Soxhlet extraction
ExtractionMethod	TOTAL	Digestion of unfiltered waters for total metals analysis
ParamID	ACE	Acetone
ParamID	AS	Arsenic
ParamID	BHCGAMMA	gamma-BHC (Lindane)
ParamID	BZ	Benzene
ParamID	CDS	Carbon disulfide
ParamID	PB	Lead
ParamID	PHENOL	Phenol
ParamID	SE	Selenium
ParamID	TCE	Trichloroethene

ATTACHMENT 2

NIROP Fridley Data Quality Objectives Notes

NIROP FRIDLEY DATA QUALITY OBJECTIVES
ANOKA COUNTY PARK VEGETABLE OIL PILOT STUDY

Notes:

- ξ This version of the notes represents all changes made in accordance with meetings and telephone discussions up to and including the 08-21-01 teleconference. A record of meetings and teleconferences is provided in Attachment 1.

- ξ The partnering Team (PT) agrees that development of Data Quality Objectives (DQOs) is important to focus the planning effort and to obtain as much agreement as possible concerning the strategy and criteria for completing each investigative effort. The team also acknowledges that DQO process outputs are commonly based on assumptions that could be invalid or the DQOs themselves could be flawed, for example, because of incomplete knowledge of site conditions at the time of planning. If this proves to be the case, adjustments might be required to complete an investigation satisfactorily. Deviations from the plan should be documented and justified.

- ξ Where feasible, reference is made to existing documents to minimize the amount of detail that needed to be recorded during this meeting. To save time, already established or addressed previously, such as budget and schedule constraints, were deliberately omitted unless they would have bearing on the development of strategies for solving the problems.

- ξ Quality control (QC) samples (type, number, etc.) will be specified in accordance with Environmental Protection Agency (EPA) and Minnesota Pollution Control Agency (MPCA) guidance in the Long Term Monitoring (LTM) work plan (WP).

- ξ The DQOs began with a review of past activities and a discussion of the problem to be solved at Fridley. It was quickly determined that there is more than one problem. The problems were separated into problems A, B, C, and D. If more problems are identified at a later date, they will be labeled alphabetically as was done for the first four problems. The notes in this document begin with a statement of general DQOs and are followed by the DQOs specific to Problem A, Anoka County Park Vegetable Oil Pilot Study. The DQOs for the other problems are provided in separate documents.

- ξ Formatting and renumbering of attachments necessary to render this set of DQOs self-consistent was conducted by T. Johnston (TTNUS) after the notes were approved on 08-21-01. Changes made were minimal and do not affect the technical content beyond the approved changes in the 08-21-01 teleconference.

DQO Step 1. State the Problem (General Section)

Conceptual Site Model (CSM): The Naval Industrial Reserve Ordnance Plant, Fridley (NIROP) has been located in the northwestern portion of the Minneapolis/St. Paul Metropolitan area, in the city of Fridley, Minnesota since 1941. The NIROP facility was the first government-owned, contractor-operated (GOCO) facility. The facility is divided into two (2) areas on the basis of ownership. The government owned NIROP site (NIROP) consists of approximately 80.3 acres to the north. This area consists of the ordnance manufacturing building and the property to the north of the building, known as the "North 40." The NIROP is contiguous and adjacent to buildings and property just to the south, owned and operated by United Defense, Limited Partnership (UDLP). The UDLP property is 55.4 acres. Anoka County Riverfront Park (ACP) to the west along the Mississippi River consists of approximately 59.8 acres. The NIROP site is situated 30 feet above and 800 feet east of the Mississippi River, and approximately 4000 feet up river from the Minneapolis Water Works river intake. Past disposal activities on the NIROP facility have resulted in soil and ground water contamination. There is trichloroethene (TCE) contamination in NIROP

upgradient wells (shallow, intermediate, deep and bedrock aquifers) indicating contamination may be coming onto the NIROP site. Ten-ppb contour lines were drawn for shallow/intermediate and deep zones:

Shallow = 20' - 30' (approximate depth)
Intermediate = 30' - 80' (approximate depth)
Deep = 80' - 120' (approximate depth)
Bedrock = 120' - 180' (actual depth)

Four extraction wells were installed in 1992 and two additional wells were installed in 1995 for hydraulic containment of the TCE plume (capture and containment). Extra wells will be added this year (2001) to prevent apparent "blow-by" of TCE past extraction wells. The area of contamination beyond the capture well system is not dissipating as expected. The Minneapolis Water Works downstream of the site on the Mississippi River has experienced a maximum TCE concentration of 1.9 ug/L. The UDLP has a plume and extraction wells on their property, also. Regulators are working with UDLP to get them up to the same level as the Navy with regard to water treatment/containment.

Pages 1 and 2 of he Record of Decision (ROD) state:

> "This action addresses the principal threat posed by the NIROP by preventing endangerment of public health, welfare, or the environment by implementation of this Record of Decision through hydraulic containment and recovery of all future migration of contaminated ground water from the NIROP and by recovery, to the extent feasible, of contamination downgradient of the NIROP."

Also on page 2, the ROD speaks of "effective" hydraulic containment in the context of " ...hydraulic containment and recovery of all future migration of contaminated ground water from the NIROP..." The ROD goes on to say on page 3:

> "A portion of the aquifer within the Anoka County Parkland closest to the Mississippi River may not fall within the zone of capture of the ground water recovery system. However, should this occur, contaminants in any uncaptured portion of the aquifer are expected to dissipate by natural means over time to levels that are protective of human health and the environment..."

The five-year review speaks of "substantial" containment. This term was used in the five-year review to reflect that "total" (i.e., 100%) hydraulic containment, as required by the ROD, is not likely to be provable. The meaning of "substantial" is not agreed upon. Total containment is interpreted by the partnering team as to not knowingly leave blow-by. How this is determined is addressed under Problem B, Effectiveness of Well Capture System.

Issue: How to define the degree of containment necessary to achieve substantial hydraulic containment must be decided.

Based on an overly conservative analysis, which has since been deleted from Annual monitoring reports, it was once estimated that up to 1 ton per year of TCE was flowing into the Mississippi River. The Navy extraction wells have removed a total of 12.5 tons of TCE between 1992 and 1999

Based on the five-year review, a recommendation was made to evaluate whether a remedy existed for the Anoka County Park (ACP) plume, and to evaluate the remedy (pp. 8 and 9 of the 5-yr review report).

At the 5-yr Review the following objectives were reiterated from the ROD:

> "Installation and operation of a groundwater containment and recovery system to hydraulically contain TCE contaminated groundwater to prevent further migration and to ultimately restore groundwater quality in the aquifer to MCLs. Installation and operation of a groundwater containment and recovery system to recover, to the extent feasible, TCE contaminated groundwater beneath Anoka County Park."

The Five Year Review goes on to recommend the following related to ACP:

- ξ "...
- ξ ...

- ξ The Navy will determine if any potential sources of contamination exist in Anoka County Park that may impact residual groundwater contamination levels in the area where residual groundwater contamination is present by September 1999.
- ξ ...
- ξ The Navy will determine what can be done to promote reduction of residual groundwater contamination in Anoka County Park to a level that will significantly reduce residual groundwater contamination, and determine if a response action will enhance the effectiveness of the selected remedy as it relates to residual groundwater in Anoka County Park by September 1999, and if warranted, will conduct a response action that will significantly reduce residual groundwater contamination and enhance the effectiveness of the selected remedy as it relates to residual groundwater contamination from NIROP in Anoka County Park by September 2000."

Note the emphasis on contamination *leaving NIROP* as opposed to *entering the river.*

Assumption: Preventing NIROP groundwater contamination from leaving the property is protective of the Mississippi River.

Assumption: FMC/UDLP contamination is distinct from NIROP contamination, even though it could be difficult to separate the two.

Assumption: Soil is outside the scope of this groundwater (GW) operable unit (OU).

Assumption: The Prairie du Chien aquifer is not contaminated.

Note: There are four basic problems and hence four different decision statements. From this point on, each problem is dealt with individually, beginning on the next page. The problems are as follow:

1. **Problem A** Anoka County Park Vegetable Oil VOC Reduction Pilot Study
2. **Problem B** Effectiveness of Capture Well System and Capture Zone Analysis
3. **Problem C: Groundwater Monitoring for** Overall Contamination at NIROP (i.e., LTM)
4. **Problem D:** Exit Strategy (identified during the July DQO meeting)

The Anoka County Park Problem is presented here. The other problems are presented in separate DQO notes, one set for each problem.

Consensus #1 (C1): The DQO notes will not be meeting minutes. The DQO notes will be an ongoing (work in progress) document which documents the meeting's decisions.

C2: Work of day will be drafted after meeting and reviewed at beginning of the next morning. Have a summary comments at the end of each am and pm. The team will recap the meeting's decisions at meeting closure to ensure we get team buy-in on site.

C3: Be trustful of one another.

C4: Changes to DQO notes. Footnote on bottom of page, date it was approved for entire document.

Action Item #1 (AI #1): Team will be notified via email if there is a conference call scheduled to address an action item. Team members will decide if they will be involved.

AI2: Initiating party of conference call (AI #1) will notify all involved parties that a conference call will take place (including date, time, topic).

Problem A: Anoka County Park (ACP) Vegetable Oil Volatile Organic Compound (VOC) Reduction Pilot Study

DQO Step 1. State the Problem

Conceptual Site Model of Anoka County Park:

MS-46S had [TCE] ~ 18,000 ppb. Vegetable oil treatment process reduces trichloroethene (TCE) by degrading to dichloroethene (DCE), then vinyl chloride (VC is more of a health threat than TCE). These are eventually further degraded to ethenes.

The contaminated GW is treated in a given location (injection zone) and as it moves downgradient the degradation products, DCE and VC are generated from TCE. TCE concentrations should decrease with distance from the treatment zone. [DCE] and [VC] should increase downstream of the treatment zone but they should eventually decrease in concentration through further degradation by the time the treated water zone has reached the contingency wells.

We wish to determine whether the treatment is effective enough to be worth extending to other portions of ACP with an objective of reducing contaminant concentrations at ACP to acceptable levels. Consideration will also be given to extending the use of the technology to treatment of NIROP building contaminant source areas. This pilot study of the vegetable oil injection technology is not required to reduce contaminants to nondetectable concentration levels, or even maximum contaminant levels (MCLs) within the time frame of the study. However, it should show promise for causing significant reductions in contaminant concentrations that could be further reduced, either by continued treatment using this same technology or by other means.

Note: There is a concern that VC generated during degradation of TCE and DCE may get to the river.

The pilot scale well configuration is presented in Attachment 2.

Past Data:
Ambient well fluctuation has been difficult to assess. Changes in sampling methods is suspected of being a contributing factor to the observed fluctuations.

DQO Step 2. State the Decision

Study Question: Is the ACP pilot study successful in significantly reducing groundwater contamination in the ACP shallow aquifer?

Potential Actions:
If successful:

1. Expand the use of the pilot study technology to the rest of the ACP
2. Consider using the technology in the NIROP source areas

If not successful, forego a larger scale implementation of the treatment technology and evaluate other remedies for reducing the groundwater contamination

Decision Statement:
Determine whether the vegetable oil pilot test for ACP has significantly reduced groundwater contamination from NIROP.

If it has, then declare the test a success and develop objectives for full-scale implementation of the technology across ACP.

If it has not, then evaluate other remedies.

Assumption: The technology may be considered for use in the NIROP source area in the future.

DQO Step 3. Inputs to the Decision

Baseline condition (ambient contaminant concentrations) and parameters: need to establish baseline condition as benchmark for determining when acceptable cleanup is achieved (if the definition of success is in terms of absolute concentrations after treatment).

Need to determine mean and variation for ambient conditions if we will measure contaminant concentrations relative to ambient concentrations.

Note: One round of sampling to establish ambient conditions is acceptable for each contaminant.

Analytes: Started with "all" VOCs; ended up with TCE, DCE, and VC; geochemical parameters as presented in the Pilot Scale Work Plan. Will monitor *cis*- and *trans*-1,2-DCE isomers.

Analytical methods: as presented in pilot scale QAPP as approved by EPA and MPCA.

Sampling methods: The purge and sample method will be used. This method is consistent with MPCA approved "Superfund and Voluntary Investigation and Cleanup Program, Sampling Protocol Template for Monitoring Wells" (July, 2001). This will also be approved by EPA.
Assumption: Detection Limits must achieve regulatory limits (the HRL limit or MCL or surface water standard, whichever is lowest, for each respective COC).

Action Levels:

Note: Percent contaminant reduction and absolute final contaminant concentrations were considered as measures of performance. Also considered was whether [TCE], [*cis*-1,2-DCE], [*trans*-1,2-DCE], and [VC] all need to have goals (reference remedial technology vendor's performance goals). It was agreed that all four chemicals should have goals.

Technology Vendor (Parsons) Performance Claim (Todd Wiedemeyer):
50% reduction in TCE concentration in at least one of the three immediately downgradient wells (MW1, 6, and 7) within 6 months. At any of the contingency wells (CW1, 2, and 3) VC and DCE will not be above ambient levels within 6 months.

Note: The achievable % reduction in contaminant concentration will be sensitive to the initial contaminant concentration.

Discussion ensued about whether the team would consider attainment of the vendor performance claim to be a success. Consideration was given to a 50% reduction of 18,000 ppb leaving a [TCE] of 9,000 ppb in the GW. The group agreed that a % reduction does not necessarily lead to a reasonable final concentration because it depends on the starting concentration.

The actual action levels are presented in DQO Step 5.

Notice was taken of the fact that DCE and VC concentrations should not increase to unacceptable levels whereas TCE concentrations must decrease to less than the action limit for the test to be successful.

DQO Step 4. Define the Study Area Boundaries

Geographical boundary: as stated the ACP pilot study work plan. Action levels must be linked to measurement position in the pilot study treatment zone.

Assumption: The selected pilot study location is representative of the rest of ACP. Therefore, performance in this zone will represent performance across the ACP.

Population of interest:

Contaminated groundwater in shallow aquifer at location of pilot study; 10-foot screen top of screen about 30' depth.

Assumption: TCE, DCE, and VC move essentially in unison in the shallow GW plume.

Note: TCE reduction occurs in the aquifer closest to the injected vegetable oil, in part, because of dissolution into the vegetable oil used in the treatment process. Therefore, the [TCE] reduction will be measured in wells MW 1, 6, or 7. [VC] and [DCE] are generated downstream of the injection zone so their concentrations will be measured in wells 27S, 47S, CW -1, CW-2, and CW-3. The CW-1 and CW-3 wells will be spread out from their originally planned positions (as discussed in workplan revisions) to better fill in the spaces between 27S and CW-2, and between CW-2 and 47S. This is expected to facilitate interception of the treated water plume. Because the wells will be spread out and there could be variations in flow from well to well (i.e., the treated water plume is not equally represented among all five wells), evaluation of [VC] and [DCE] trends will be done for each well.

Temporal Boundary:

Period of performance: 0 year <= time <= 2 years.

Decision time frame: Decision will be made within 2-yr time frame.

Decision will be based on comparison of before treatment and after treatment concentrations of target analytes.

Issues:

- ξ Remobilization of VOCs could occur near the river because high levels of adsorbed VOC re-equilibrate with cleaner water coming from the treatment zone.
- ξ Additional extraction wells could cause contaminant reduction in pilot test area.
- ξ Additional extraction wells could affect potentiometric surface in pilot test area.

DQO Step 5. Develop a Decision Rule

Note: VOC_{select} = TCE, *cis*-1,2,-DCE, *trans*-1,2-DCE and VC

Assumption: The treatment technology may be considered for use by the partnering team in NIROP source areas (i.e., underneath building). There are many factors that will affect this decision. The partnering team does not have enough information to address this more definitively at this time.

Baseline (i.e., ambient) concentrations and associated variability were established for DCE and VC by performing statistical analyses on Total DCE and VC data from 1997 to 1999 in wells 27S and 47S. These wells are two of the five contingency wells that will be used for monitoring *cis*- and *trans*-1,2-DCE and VC and they bracket the entire array of five contingency wells. There are no VOC data available from the other three contingency wells (CW-1, 2 and 3). There were not enough *cis*-1,2-DCE or *trans*-1,2-DCE data for statistical analyses so the total DCE results were used as a surrogate for *cis*-1,2-DCE and *trans*-1,2-DCE.Results of the statistical analyses (years 1997 to 1999) and the computed action limits are shown in the next table. The action limits and uncertainties shown are expected to be "worst case" values because they are based on data that span a 3-year period.

Statistical Analysis and Action Levels

Analyte	Well 27S	Well 47S	Overall Ambient	Regulatory Limit	Action Level[4]
Total [1,2-DCE][1]	21 ρ 11 ppb	29 ρ 1 ppb	20 ρ 15 ppb[2]	70 ppb[3]	90 ppb
Cis - 1, 2- DCE	ID	ID	ID	70 ppb	ID
Trans - 1,2- DCE	ID	ID	ID	100 ppb	ID
VC	ND	ND	ND	0.18 ppb[3]	0.3 ppb
Trichloroethene	143 ρ 23[5]	60 ρ 4	125 ρ 45	NA	1,000 ppb

(1) Used to represent *cis-* and *trans-1,2*-DCE because not enough data are available to compute results for *cis- and trans*-1,2-DCE, individually.
(2) Number in this column represents worst case average conditions rounded down to nearest 5 units for mean value and rounded up to nearest 5 units for standard deviation (1-sigma). The variability affects the "number of samples" calculation. Greater variability leads to needing more samples.
(3) MCL
(4) Mean value plus regulatory limit in the pilot scale study area, rounded to one significant figure.
(5) The temporal data showed a clear downward trend (about 1,700 ppb down to about 150 ppb) over the 1997-1999 time frame, and the trend leveled off over the last three data points. The values shown are based on the last three rounds of data.

ID - insufficient data at this time. As baseline data are generated, these numbers will be inserted.

Note: T. Johnston (TtNUS) has verified with Paul Walz of Bay West (651-291-3491) that the standard approach is to report results from both diluted and undiluted sets of results. Furthermore, we should be able to achieve a detection limit of 0.17 ppb VC in all samples that have less than about 2,000 ppb TCE. Samples with more than about 2,000 ppb TCE in them would have to be diluted to prevent instrument down time, or additional analytical costs would be incurred for cleaning the high levels of TCE out of the analytical instruments and/or replacing damaged parts.

Decision Rules:

If [TCE] is reduced to < 1000 ppb AND the temporal [TCE] trend is not increasing in one or more of wells MW 1, 6, or 7 AND if [VC], [*cis-1,2*-DCE] and [*trans-1,2*--DCE] are <= (the COC's regulatory limit + current ambient concentrations) in CW 1, 2, and 3 and 27-S and MS-47S within the 2 year pilot study time frame, *then* declare success and develop objectives for full scale implementation of the technology across ACP.

If [TCE] is not reduced to < 1000 ppb OR the temporal [TCE] trend is increasing in one or more of wells MW 1, 6, or 7 OR if [VC], [*cis-1,2*—DCE] and [*trans-1,2*--DCE] are not <= (the COC's regulatory limit + current ambient concentrations) in CW 1, 2, and 3 and 27-S and MS-47S within the 2 year pilot study time frame, then reevaluate the adequacy of the pilot study and/or evaluate alternate remedies.

If [TCE] is reduced to < 1000 ppb AND the temporal [TCE] trend is not increasing in one or more of well MW 1, 6, or 7 BUT the [VC] or [*cis-1,2*—DCE] or [*trans-1,2*--DCE] > (the COC's regulatory limit + current ambient concentration) in wells 27S, 47S, CW-1, CW-2, or CW-3 within the 2-year pilot study time frame, OR the [VC] or [*cis-1,2*--DCE or *trans-1,2*--DCE] trend is projected to increase to a level that is greater than the action level within five years, *then* consult the partnering team to decide whether implementation of Parsons' contingency plan is required.

Note: These decision rules are presented in flow chart form in Attachment 3. The use of TCE, VC, *cis*-1,2-DCE and *trans*-1,2-DCE may be clearer in the flow charts.

On the assumed basis that the downgradient wells (27S, 47S, CW-1, CW-2 and CW-3) results will not be averaged for each sampling round, a sampling strategy was developed to fit the DQOs. The strategy is provided in Attachment 4.

DQO Step 6. Establish Error Tolerances

This step was not implemented. Instead, Tom Johnston (TtNUS) and B Lewis (TtNUS) worked up a temporal sampling strategy based on the above DQO outputs. The development of that strategy is described in Attachment 4.

Note: The sampling plan described in Attachment 4 is consistent with the conversation held with Todd Weidemeyer (Parsons). The strategy was described briefly to Todd Weidemeyer (Parsons) by Tom Johnston (TtNUS) on April 2, 2001. Todd confirmed that only the well showing the greatest reduction in TCE concentration should be used for data trending. Data from all wells will be evaluated and discussed in the pilot study report, but only the well with the greatest TCE reduction will be used to evaluate the pilot scale study success.

Note: Statistical basis could require more than the four sampling points already selected.

DQO Step 7. Optimize the Design
A sampling strategy is laid out in Attachment 4. Sampling locations are shown in the ACP Pilot Study work plan.

Attachment 1. DQO Meeting Record, 2001

DQO Meeting Attendees:

		Meeting Dates						
Name	**Affiliation**	**3/21 - 3/24, C[3]**	**5/24, T**	**7/12, T**	**7/17-19, F**	**8/21, T**		
Joel Sanders	SOUTHDIV	X			X	X		
Thomas Bloom	USEPA	X			X[4]	X		
Hal Davis	USGS	X			X			
Keith Henn	TtNUS	X	X		X	X		
Mark Sladic	TtNUS	X	X		X	X		
Cliff Casey	SOUTHDIV	X			X			
B. Venky Venkatesh[1]	CH2MHILL	X	X		X	X		
Brian Lewis	TtNUS	X		X		X		
John Betcher[2]	MPCA	X			X	X		
David Douglas[2]	MPCA	X			X	X		
Rick kuhlthau[1]	Techlaw	X			X			
Tom Johnston	TtNUS	X	X	X	X	X		
Paul Rice	Galileo				X			
Todd Weidemeyer	Parsons		X					
Dan Griffiths	Parsons		X					
Mike Trojan	MPCA			X				

[1] Not present in the DQO meeting on Friday, March 23, 2001.

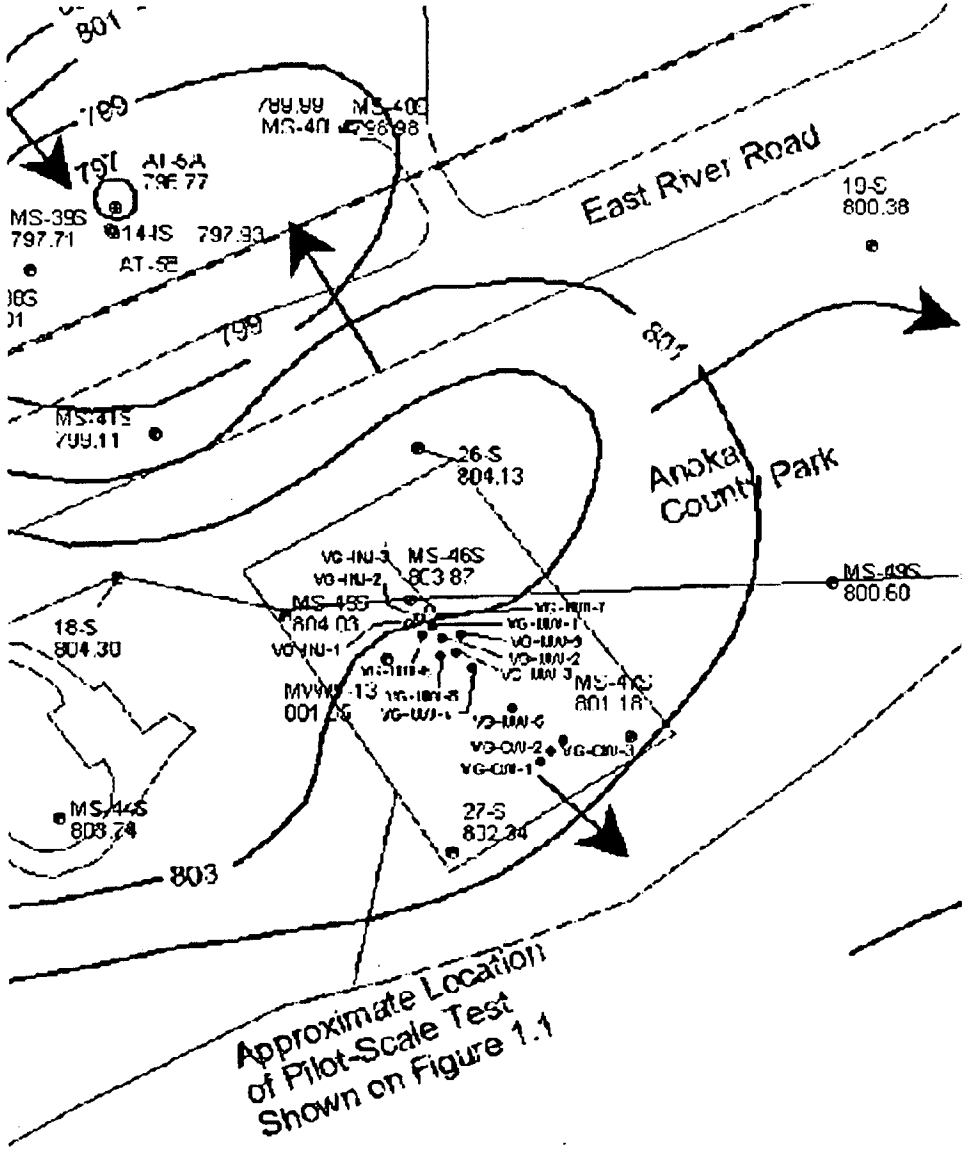
[2] Left the DQO meeting on Friday, March 23, 2001 after addressing their comments. The meeting lasted about half an hour beyond that point to address comments of other meeting participants.

[3] The first day of this meeting was a day of DQO training presented by John Warren and Tom Dixon of EPA headquarters.

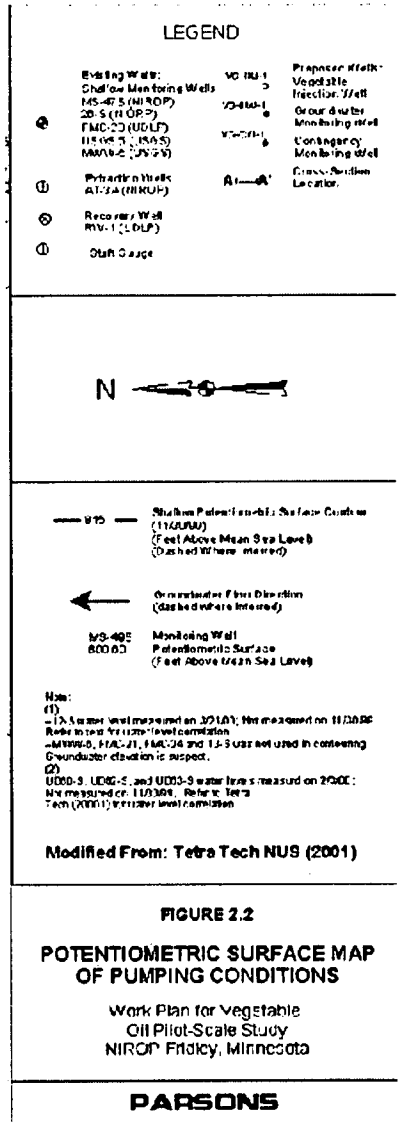
[4] Arrived late on Tuesday.

C Charleston, SC
F Fridley, MN
T Teleconference

Attachment 2. Veg. Oil Treatability study well configuration .

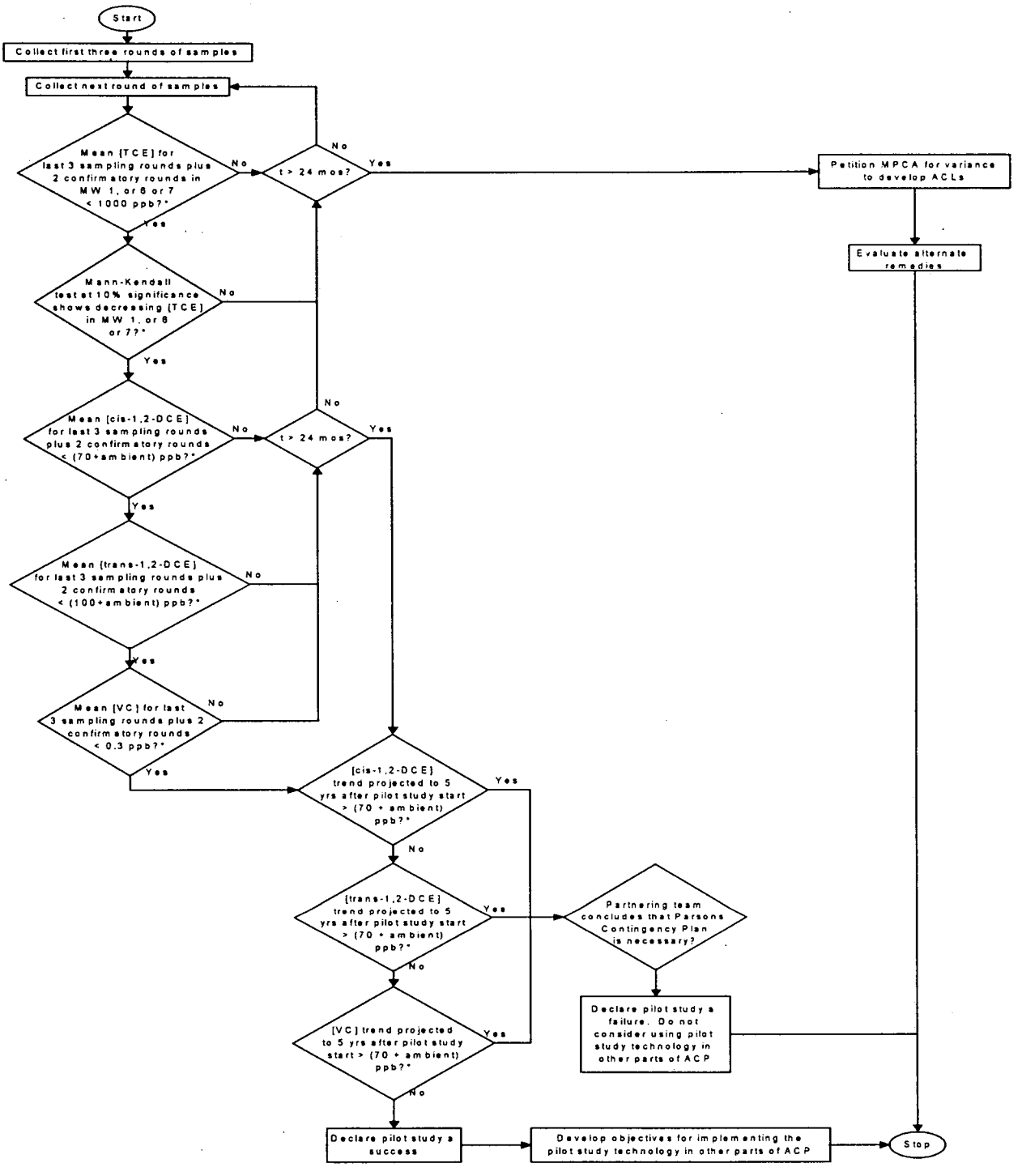


(Excerpt from Parsons Vegetable Oil Pilot Study Work Plan, Figure 2.2) Legend below:



Approved 8-21-01.

Attachment 3. Data Use Decision Logic



* [TCE] is to be measured in the well selected from CW1, CW2, CW3, 27S and 47S that shows the greatest [TCE] reduction; [cis-1,2-DCE], [trans-1,2-DCE], and [VC] are to be measured in all downgradient wells (CW1, CW2, CW3, 27S and 47S).
ACL = Alternate concentration limit
cis-1,2-DCE = cis-1,2-dichloroethene
trans-1,2-DCE = trans-1,2-dichloroethene
VC = Vinyl chloride

Attachment 4. Sampling Strategy for Problem A: Anoka County Park (ACP) Volatile Organic Compound (VOC) Reduction Pilot Study

Introduction

With the help of Mike Trojan (MPCA) and Arthur Lubin (EPA Region 5), Tom Johnston (TtNUS) and Brian Lewis (TtNUS) developed a more detailed sampling approach to deal with the TCE concentration data to be collected over time during the ACP pilot study. That approach is presented here. It deals with not only an expected decreasing concentration trend but also accommodates uncertainty in the data by using a moving window average, which is explained below. First, the proposed data use is presented, then the rationale for the proposal is presented.

Sampling Design (TCE, *cis*-1,2-DCE, *trans*-1,2-DCE, and VC)

Eight sampling events are proposed over the two year pilot test evaluation period: 0 months, 2 months, 5 months, 8 months, 12 months, 16 months, 20 months, and 24 months. The spacing of sampling times is closer together early in the pilot study to account for an expected rapid decrease in TCE concentrations. More frequent sampling is necessary for defining a trend when concentration changes are more rapid. A three-point moving average is recommended (justification found below) as a means of quantifying the TCE , *cis*-1,2-DCE, *trans*-1,2-DCE, and VC concentrations during these evaluations. The multifold criteria for success are presented in the next section. This proposed sampling design should allow a conclusion of pilot test success to be drawn as early as possible during the pilot scale test while providing reasonable confidence in the conclusion.

Criteria for Success (TCE)

The pilot study will be considered a success if all of the following criteria are met within the two-year pilot test time frame:

1. The trichloroethene (TCE) concentration median over the three most recent sampling events is less than 1,000 ppb in at least one of the three monitoring wells: MW1, MW6, and MW7;
2. The Mann-Kendall test for trends results in a conclusion that a downward trend (negative slope of trend line) exists at 90% confidence. 90% confidence was selected as a reasonable level of confidence consistent with the rigor of the study;
3. Concentrations of TCE biodegradation products, *cis*- and *trans*-1,2-dichloroethene (*cis*- and *trans*-1,2-DCE) and vinyl chloride (VC), do not exceed their current ambient concentrations plus the respective regulatory limit in contingency wells CW1, CW2, CW3, 27S, or 47S within the pilot study time frame. If the VC OR *cis*-or trans-1,2-DCE concentration in any of the five contingency wells is greater than the ambient concentration plus the regulatory limit, OR if the VC OR cis- or *trans*-1,2-DCE concentration trend projected to five years past the pilot study start date exceeds the action level, the Partnering Team will discuss whether implementing the contingency plan is necessary.

4. The concentration of each analyte is less than its action level based on an analysis of all data obtained for the first sampling event for which the moving window average of TCE balls below 1,000 ppb PLUS two consecutive sampling events thereafter. That is, after the moving window [TCE] falls below 1,000 ppb, two more sampling rounds will be conducted to verify that contaminant concentrations are staying below 1,000 ppb.

Calculation of Action Levels

Action levels for the analytes to be measured are established as the analyte's regulatory limit plus the ambient concentration of the analyte. Monitoring well data from 1997 through 1999 for wells 27S and 47S were used to compute ambient concentrations. Action levels are presented in Problem #1 DQOs, Step 5. The action levels for *cis*- and *trans*-1,2,-DCE are undetermined at this time because insufficient data were available to compute the ambient concentrations of those analytes. Additional data must be collected to compute the ambient concentrations.

The following reasoning was used to determine how many rounds of data are required to estimate the ambient *cis*- and *trans*-1,2-DEC concentrations. The concentration of neither *cis*-1,2,-DCE nor *trans*-1,2-DCE can be greater than the concentration of total 1,2-DCE (21 ppb). The ambient concentration of total 1,2-DCE (21 ppb) is relatively small — approximately one-fourth the *cis*- and *trans*-1,2-DEC regulatory limits (70 ppb and 100 ppb, respectively). The standard deviation of the total 1,2-DCE ambient concentration (11 ppb) is also relatively small. The year 2000 data for these analytes is comparable. Therefore, it would be logical to use *cis*- and *trans*-1,2-DCE concentrations from the first round of sampling and the year 2000 sampling to establish the action levels (ambient concentration + regulatory limit) for both *cis*- and *trans*-1,2-DCE. The 2000 results for *cis*- and *trans*-1,2-DCE are presented here:

Sample	Sample Date	Parameter	Result, ug/L	QC Type	Original Sample
MS-47S	10/10/00	CIS-1,2-DCE	9.9	NA	
MS-47S	10/10/00	TRANS-1,2-DCE	5.6	NA	
MS-27S	10/12/00	CIS-1,2-DCE	6.2	NA	
QCD04	10/12/00	CIS-1,2-DCE	6.2	Duplicate	MS-27S
MS-27S	10/12/00	TRANS-1,2-DCE	1 U	NA	
QCD04	10/12/00	TRANS-1,2-DCE	1 U	Duplicate	MS-27S

Justification for a Three-Point Moving Median (TCE)

Two approaches were originally considered for estimating whether pilot test success is achieved. Primary emphasis was placed on the need to determine when TCE concentrations are less than 1,000 ppb. One approach considered was a comparison of confidence bounds around a temporal [TCE] decay trend to the 1,000 ppb TCE action level. Both linear and exponential decay models were considered. The second approach was a comparison of a moving average TCE concentration to the 1,000 ppb TCE action level. The confidence bounds approach was very unpredictable, in part, because the shape of the TCE decay

function is unknown. That uncertainty, which adds to the random data uncertainties, resulted in a requirement to collect numerous samples. The number of data points required to satisfy this approach was judged to be artificially elevated and unwarranted because of the uncertainty in the decay function shape.

The moving average approach was selected as the more viable option. This option also provides a simpler mathematical approach than the confidence bounds strategy. A moving average is a data smoothing technique that establishes the mean value of a set of most recently acquired data values. As each new data point is acquired, the earliest data point in the window is discarded and the newest point is used to compute the mean. Including a larger number of values within the window yields a greater degree of smoothing. When rapid changes in concentration must be measured as a function of time, a large window tends to average out the rapid change and to show a much less rapid change. Conversely, including only a few data points within the window has a lesser smoothing effect. It aids in detecting rapid changes but tends to limit the ability to distinguish a concentration difference between the data and a numerical action level because of data scatter. The goal is to select a window size that will: (1) detect a steep trend while (2) maintaining the ability to discriminate between a numerical action level and the data average. If the data function (in this case, a temporal concentration decay function) is unknown or the magnitude of the data uncertainty is unknown, this optimization will depend more on professional judgment than on numerical computations.

Some limited data from groundwater monitoring wells was discussed during the DQO meetings. Any random uncertainty (2-sigma) at concentrations between 500 ppb and 1,000 ppb are estimated to range from 0 ppb to approximately 500 ppb. Using a single point concentration estimate, a maximum 500 ppb error would lead to a maximum value of 1,500 ppb TCE being judged as a success (assuming the other success criteria are also satisfied) even though 1,000 ppb is the action level. With a moving window average the chance of declaring a final concentration as great as 1,500 ppb to be a success is reduced further. It is believed that an error of this magnitude is tolerable given that other success criteria must satisfied simultaneously. It is believed that, if the test is successful, [TCE] is likely to be decidedly less than 1,000 ppb. So, a statistical strategy including a quantification of random errors was judged to be unnecessary.

A goal used in developing the mathematical data summary requirements was to provide the earliest possible conclusion of test success or failure while providing reasonable confidence in that conclusion. This required a compromise between the number of time points to be included in the moving window and the level of confidence obtained in the assessment. The level of decision confidence was not quantified for reasons described above.

A three-point moving average was selected as a reasonable compromise because:

1. At least three events are needed to evaluate a temporal trend and the associated uncertainties. Furthermore, at least three points are required to establish a nonlinear trend which is the expected general shape of the TCE decay function.
2. Including a third sampling event in the moving average facilitates the assessment of whether TCE concentration rebounding occurs (TCE concentrations begin to increase after an observed decrease). Admittedly the ability to detect rebound with only three data points is limited, but the partnering team would have opportunities to collect additional data before declaring success or failure if data trends are inconclusive. Discussions with Mike Trojan of the MPCA led to the conclusion that using the Mann-Kendall test for trends would help in this regard.
3. At least two samples are needed to evaluate uncertainty in concentrations due to random error when measuring any fixed concentration (e.g., when the slope of a temporal trend is equal to zero). If the mean concentration for the last three sampling events is less than 1,000 ppb, even greater confidence is provided that the true mean concentration is less than 1,000 ppb than if only the last one or two sampling events was considered. Based on rough uncertainty estimates of historical TCE data, three points are viewed to be necessary to address this concern for concentrations between 500 ppb and 1,000 ppb.
4. The selected width (three points) of the moving time window would not span such a long time frame that a declaration of success is delayed unnecessarily. For example, even if the test is overwhelmingly successful (e.g., [TCE] plummets to less than 1000 ppb within 2 months), a declaration of success may be delayed for four months if the moving average time window is expanded to include a fourth data value.

Justification for a Three-Point Moving Median and projected Trend (VC and cis- and *trans*-1,2-DCE)

The justification for using a moving average for these two analytes is similar to the justification for the TCE moving window median. Use of a 5-year projected concentration trend protects against VC or cis-or *trans*-1,2-DCE concentrations that keep increasing, even after completing the pilot study. It is assumed that concentrations of these analytes will not increase indefinitely and that, if the projected concentration trend does not exceed the action limits after five years from the start of the test, the concentrations are not likely to exceed the limits after beyond years.

"Validation" of the Number of Points in a Moving Window

It was decided that an independent test of the number of sample points included in the moving window would be useful. For this, EPA's DQO software, Decision Error Feasibility Trial (DEFT), was used to compute the number of samples that should be averaged to determine whether the mean [VC] or [*cis*-DCE] is greater than the action level. The reasoning was that, if the computed number of data points was consistent with the estimate based on professional judgment, the judgment would stand without question.

If the judgment and the number of computed data points that was computed using DEFT differed significantly, the judgment would be re-evaluated. The DEFT software is not strictly applicable to moving window averages but it provides a technical basis for estimating the number of samples required to meet decision performance specifications. The inputs to the model and the results are presented in Attachment 2. The alpha and beta levels were set at $\Delta = 10\%$ and $E = 35\%$. Alpha is the risk of thinking the action level is not exceeded when it is (i.e., incorrectly declaring success). Under these circumstances the null hypothesis is that the test is unsuccessful, and the burden of proof is on the data to demonstrate otherwise. Beta is the risk of thinking the action level is exceeded when it is not (i.e., incorrectly declaring failure). These were thought to represent reasonable risk levels for making each type of decision error, especially considering that the estimates of standard deviation (see table of statistics in DQO Step 5, Problem #1) are probably exaggerated. If the estimated standard deviation was not thought to be an overestimate, a lesser alpha value might be warranted. Given the outcome of the DQO calculations, a moving window average of three data rounds in each well is a reasonable choice. The choice of 35% false negative (E) error rate is conservative because it tends toward declaring the test a failure more readily than declaring success. However, the expected overestimate of sigma should result in actual Type II errors rates less than 35%.

Note: Additional DCE calculations (not shown) reveal that both the Δ and E error rates can be reduced to 4% or less and still only three samples are required. On the other hand, for VC, reducing E to 31% and leaving Δ at 10% increases the number of required samples to 4. This sensitivity in a small change of E indicates that, VC could be the limiting case that governs the achievable decision performance.

Inclusion of a Non-parametric test for trends

Mike Trojan of MPCA also recommended using a non-parametric test for trends to help verify the existence of a downward trend in the decay curve. Johnston (TtNUS) and Lewis (TtNIUS) agreed to this, so the Mann-Kendall test for trends will be included in the data evaluation to establish whether a downward trend exists in the TCE, *cis*-1,2-DCE , *trans*-1,2-DCE, and VC.

Expected Sampling Scheme Performance

Using the proposed sampling scheme and an expected initial TCE concentration near 15,000 ppb (discussed during DQO meetings), a minimum of four sampling events is required. Two confirmational rounds of sampling will be required thereafter. If all criteria for success are satisfied within two years of the start of the pilot test, no further sampling events need to occur and a conclusion may be drawn at that point concerning the success of the project. If the test is not declared a success, it will be considered a failure and will not be applied in other parts of ACP.

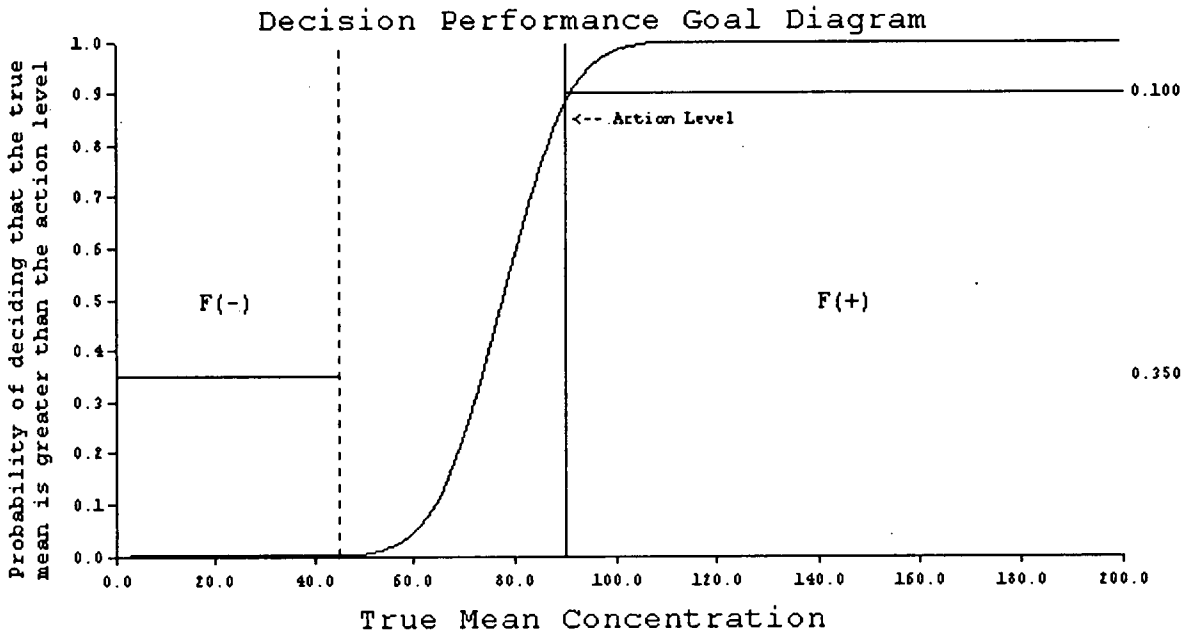
Attachment 5

DQO Calculations

Note: Disregard costs presented at the bottom of each Decision Performance Goal Diagram. Those costs are included automatically in the EPA DEFT software but were not used for these computations.

Note: Additional calculations reveal that the DEFT calculation is very sensitive to the value of the standard deviation of VC concentrations. This is partly because of the small difference between expected concentrations and the action level, which is close to the VC detection limit. the Based on these observations, the recommendation is to proceed with a three-point moving median and to expect to see essentially all non-detects for vinyl chloride. If detectable levels of vinyl chloride are observed, a weight of evidence evaluation should be implemented to determine whether the vinyl chloride is present at unacceptable concentrations.

DEFT Number of Samples Calculation
Total 1,2-Dichloroethene (surrogate for cis- and trans-1,2,-DCE), sigma = 15 ppb

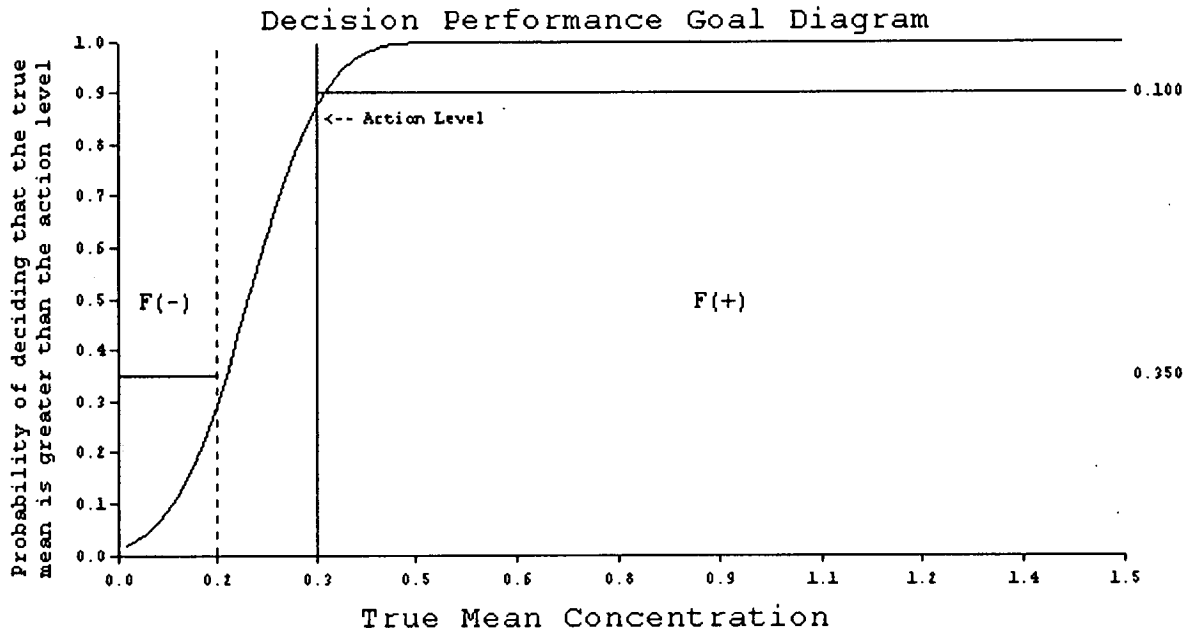


Simple Random Sampling
Action Level = 90.00
Cost = $2100.00
Sample Size = 2

Decision Error Limits

concentration	prob(E)	type
45.00	0.350	F(-)
90.00	0.100	F(+)

DEFT Number of Samples Calculation
Vinyl Chloride, sigma = 1 ppb

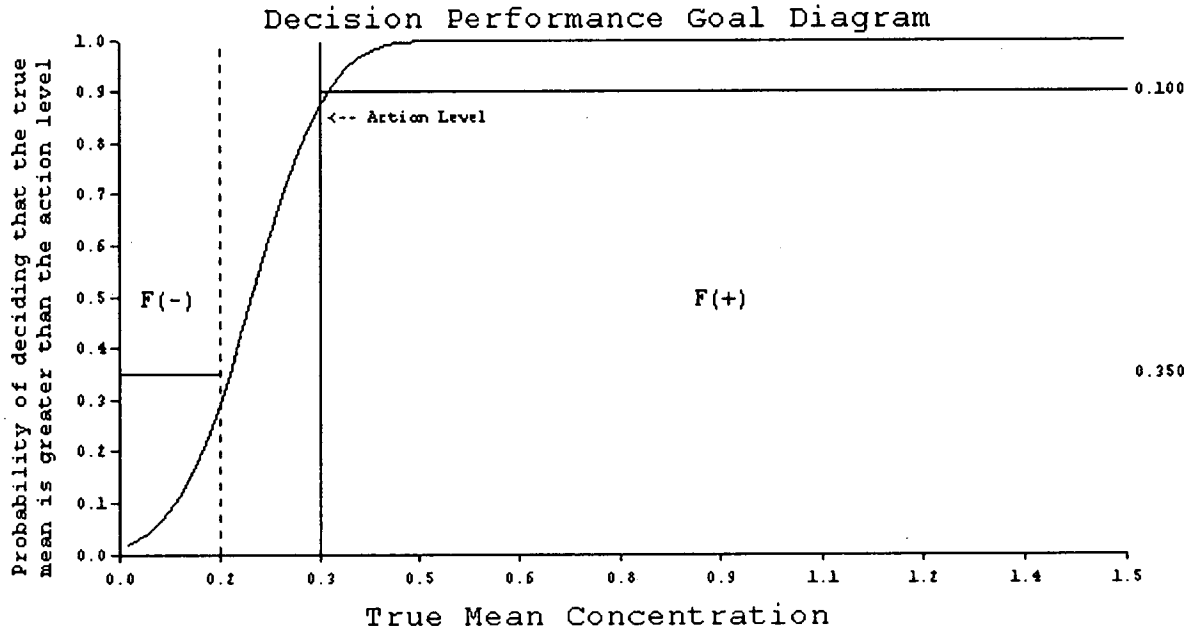


Simple Random Sampling
Action Level = 0.30
Cost = $131250.00
Sample Size = 125

Decision Error Limits

concentration	prob(E)	type
0.15	0.350	F(-)
0.30	0.100	F(+)

DEFT Number of Samples Calculation
Vinyl Chloride, sigma = 0.1 ppb

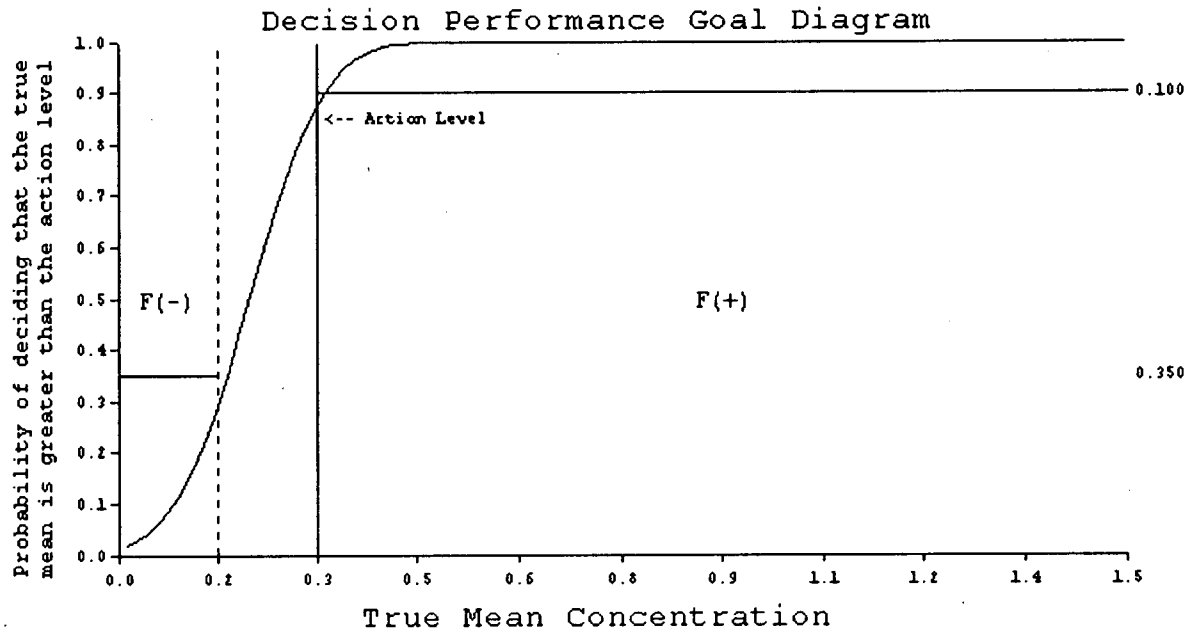


Simple Random Sampling
Action Level = 0.30
Cost = $3150.00
Sample Size = 3

Decision Error Limits

concentration	prob(E)	type
0.15	0.350	F(-)
0.30	0.100	F(+)

DEFT Number of Samples Calculation
Vinyl Chloride, sigma = 0.2 ppb

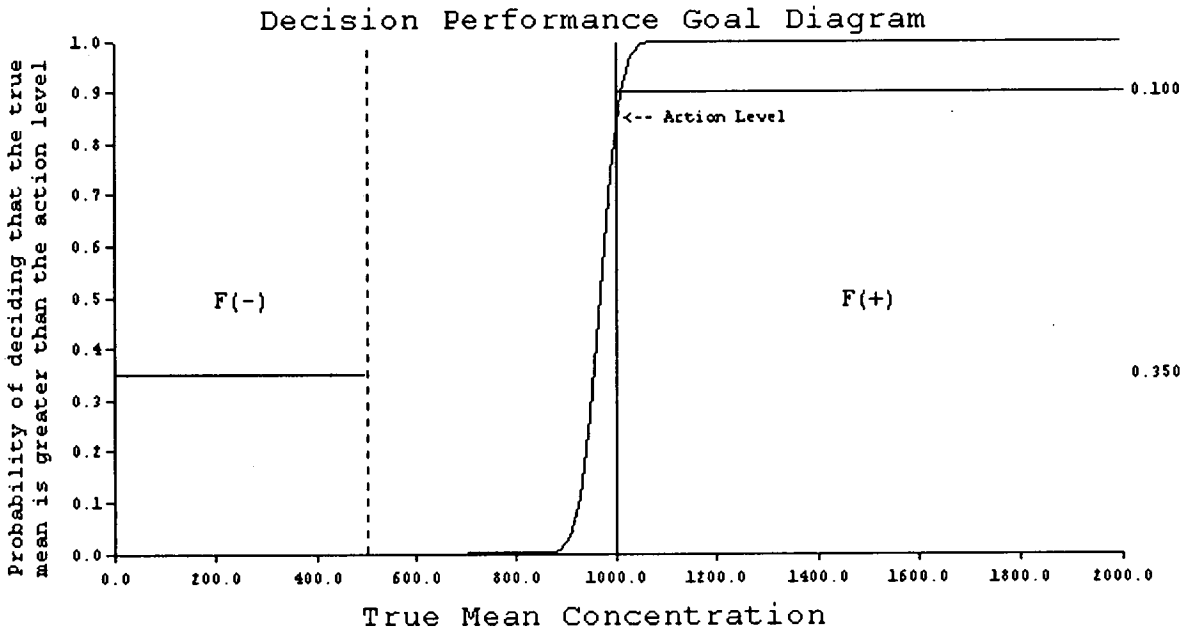


Simple Random Sampling
Action Level = 0.30
Cost = $6300.00
Sample Size = 6

Decision Error Limits

concentration	prob(E)	type
0.15	0.350	F(-)
0.30	0.100	F(+)

DEFT Number of Samples Calculation
Trichloroethene, sigma = 45 ppb



Simple Random Sampling
Action Level = 1000.00
Cost = $2100.00
Sample Size = 2

Decision Error Limits

concentration	prob(E)	type
500.00	0.350	F(-)
1000.00	0.100	F(+)

ATTACHMENT 3

ENCHEM, INC. PROJECT-SPECIFIC QUALITY ASSURANCE OBJECTIVES

EN CHEM MADISON LABORATORY ANALYTICAL METHODS, QA OBJECTIVES FOR AQUEOUS SAMPLES

Effective June 2001 to June 2002

5/4/01

Parameter	units	Method (prep)	Method (analytical)	LCS (% Rec.)	LCS (% RPD)	Accuracy (% Rec.)	Precision (% RPD)	MDL	Reporting Limit	En Chem SOP #
Volatile 8260B										
1 - Propanol	ug/L	5030	8260B	N/A	N/A	70-130	0-40	10.0	10.0	VOA-5
1,1 - Dichloroethane	ug/L	5030	8260B	83-132	0-10	83-141	0-6	0.47	1.0	VOA-5
1,1 - Dichloroethene	ug/L	5030	8260B	81-145	0-10	82-150	0-7	0.47	1.0	VOA-5
1,1 - Dichloropropen	ug/L	5030	8260B	70-130	0-40	61-169	0-9	0.38	1.0	VOA-5
1,1,1 - Trichloroethar	ug/L	5030	8260B	87-137	0-10	88-142	0-8	0.53	1.0	VOA-5
1,1,1,2 - Tetrachloro	ug/L	5030	8260B	70-130	0-40	83-129	0-6	0.49	1.0	VOA-5
1,1,2 - Trichloro - 1,2	ug/L	5030	8260B	50-150	0-50	82-149	0-7	0.61	2.0	VOA-5
1,1,2 - Trichloroethar	ug/L	5030	8260B	88-131	0-10	83-128	0-8	0.44	1.0	VOA-5
1,1,2,2 - Tetrachloro	ug/L	5030	8260B	86-132	0-10	72-144	0-12	0.64	1.0	VOA-5
1,2 - Dibromo - 3 - Cl	ug/L	5030	8260B	70-130	0-40	70-133	0-13	0.76	2.0	VOA-5
1,2 - Dibromoethane	ug/L	5030	8260B	70-130	0-40	82-126	0-9	0.39	1.0	VOA-5
1,2 - Dichlorobenzen	ug/L	5030	8260B	70-130	0-40	86-125	0-5	0.38	1.0	VOA-5
1,2 - Dichloroethane	ug/L	5030	8260B	84-138	0-10	77-137	0-7	0.52	1.0	VOA-5
1,2 - Dichloroethene,	ug/L	5030	8260B	80-124	0-13	86-138	0-15	0.73	2.0	VOA-5
1,2 - Dichloropropan	ug/L	5030	8260B	90-127	0-10	80-139	0-5	0.46	1.0	VOA-5
1,2,3 - Trichlorobenz	ug/L	5030	8260B	70-130	0-40	69-134	0-18	0.33	1.0	VOA-5
1,2,3 - Trichloroprop	ug/L	5030	8260B	70-130	0-40	80-126	0-14	0.51	1.0	VOA-5
1,2,3 - Trimethylbenz	ug/L	5030	8260B	70-130	0-40	90-126	0-5	0.37	1.0	VOA-5
1,2,4 - Trichlorobenz	ug/L	5030	8260B	70-130	0-40	71-132	0-12	0.35	1.0	VOA-5
1,2,4 - Trimethylbenz	ug/L	5030	8260B	70-130	0-40	89-129	0-7	0.27	1.0	VOA-5
1,3 - Dichlorobenzen	ug/L	5030	8260B	70-130	0-40	86-126	0-5	0.42	1.0	VOA-5
1,3 - Dichloropropan	ug/L	5030	8260B	70-130	0-40	87-125	0-6	0.42	1.0	VOA-5
1,3,5 - Trimethylbenz	ug/L	5030	8260B	70-130	0-40	89-131	0-6	0.27	1.0	VOA-5
1,4 - Dichlorobenzen	ug/L	5030	8260B	70-130	0-40	85-123	0-5	0.50	1.0	VOA-5
1,4 - Dioxane	ug/L	5030	8260B	N/A	N/A	70-130	0-40	35.95	100.0	VOA-5
2 - Butanone	ug/L	5030	8260B	59-122	0-10	44-141	0-17	1.03	5.0	VOA-5
2 - Chloroethylvinylet	ug/L	5030	8260B	70-130	0-40	N/A	N/A	0.25	1.0	VOA-5
2 - Chlorotoluene	ug/L	5030	8260B	N/A	N/A	91-128	0-5	0.25	1.0	VOA-5
2 - Hexanone	ug/L	5030	8260B	60-128	0-10	44-134	0-20	0.80	5.0	VOA-5
2 - Methylnaphthalen	ug/L	5030	8260B	N/A	N/A	70-130	0-40	0.37	5.0	VOA-5
2,2 - Dichloropropan	ug/L	5030	8260B	70-130	0-40	69-151	0-7	0.43	1.0	VOA-5

EN CHEM MADISON LABORATORY ANALYTICAL METHODS, QA OBJECTIVES FOR AQUEOUS SAMPLES

Effective June 2001 to June 2002 5/4/01

Parameter	units	Method (prep)	Method (analytical)	LCS (% Rec.)	LCS (% RPD)	Accuracy (% Rec.)	Precision (% RPD)	MDL	Reporting Limit	En Chem SOP #
2,3 - Dichloropropen	ug/L	5030	8260B	N/A	N/A	91-127	0-6	0.39	1.0	VOA-5
4 - Chlorotoluene	ug/L	5030	8260B	70-130	0-40	87-128	0-5	0.37	1.0	VOA-5
4 - Methyl - 2 - penta	ug/L	5030	8260B	67-132	0-10	64-137	0-19	0.90	5.0	VOA-5
Acetone	ug/L	5030	8260B	24-151	0-15	5-153	0-17	1.64	5.0	VOA-5
Acetonitrile	ug/L	5030	8260B	N/A	N/A	70-130	0-40	0.51	50.0	VOA-5
Acrolein	ug/L	5030	8260B	N/A	N/A	45-313	0-25	6.88	20.0	VOA-5
Acrylonitrile	ug/L	5030	8260B	N/A	N/A	66-142	0-13	0.42	10.0	VOA-5
Allyl Chloride	ug/L	5030	8260B	N/A	N/A	76-147	0-14	0.42	1.0	VOA-5
Benzene	ug/L	5030	8260B	83-128	0-10	89-138	0-8	0.44	1.0	VOA-5
Benzyl Chloride	ug/L	5030	8260B	N/A	N/A	70-130	0-40	1.34	50.0	VOA-5
Bromobenzene	ug/L	5030	8260B	70-130	0-40	87-127	0-5	0.39	1.0	VOA-5
Bromochloromethan	ug/L	5030	8260B	70-130	0-40	81-135	0-9	0.54	1.0	VOA-5
Bromodichlorometha	ug/L	5030	8260B	88-133	0-10	82-134	0-6	0.42	1.0	VOA-5
Bromoform	ug/L	5030	8260B	81-143	0-10	63-136	0-9	0.52	1.0	VOA-5
Bromomethane	ug/L	5030	8260B	48-130	0-13	34-176	0-32	0.69	2.0	VOA-5
Carbon disulfide	ug/L	5030	8260B	81-135	0-10	83-142	0-7	0.51	1.0	VOA-5
Carbon tetrachloride	ug/L	5030	8260B	72-147	0-10	79-145	0-8	0.54	1.0	VOA-5
Chlorobenzene	ug/L	5030	8260B	94-122	0-10	87-125	0-6	0.48	1.0	VOA-5
Chlorodibromometha	ug/L	5030	8260B	85-132	0-10	80-131	0-8	0.51	1.0	VOA-5
Chloroethane	ug/L	5030	8260B	72-134	0-10	79-153	0-11	0.69	2.0	VOA-5
Chloroform	ug/L	5030	8260B	86-132	0-10	86-136	0-8	0.47	1.0	VOA-5
Chloromethane	ug/L	5030	8260B	19-143	0-10	61-161	0-14	0.83	2.0	VOA-5
Chloroprene	ug/L	5030	8260B	N/A	N/A	70-130	0-40	1.01	10.0	VOA-5
cis - 1,2 - Dichloroeth	ug/L	5030	8260B	86-128	0-10	89-136	0-6	0.99	1.0	VOA-5
cis - 1,3 - Dichloropr	ug/L	5030	8260B	84-130	0-10	78-135	0-6	0.29	1.0	VOA-5
cis - 1,4 - Dichloro - 2	ug/L	5030	8260B	N/A	N/A	57-136	0-16	0.57	1.0	VOA-5
Crotonaldehyde	ug/L	5030	8260B	N/A	N/A	70-130	0-40	58.2	1000.0	VOA-5
Cyclohexane	ug/L	5030	8260B	N/A	N/A	70-130	0-40	0.43	5.0	VOA-5
Cyclohexanone	ug/L	5030	8260B	N/A	N/A	70-130	0-40	100	100.0	VOA-5
Di - isopropyl ether	ug/L	5030	8260B	70-130	N/A	73-150	0-5	0.28	1.0	VOA-5
Dibromomethane	ug/L	5030	8260B	70-130	0-40	78-133	0-7	0.59	1.0	VOA-5
Dichlorodifluorometh	ug/L	5030	8260B	50-150	0-50	72-143	0-9	0.94	2.0	VOA-5

EN CHEM MADISON LABORATORY ANALYTICAL METHODS, QA OBJECTIVES FOR AQUEOUS SAMPLES

Effective June 2001 to June 2002 5/4/01

Parameter	units	Method (prep)	Method (analytical)	LCS (% Rec.)	LCS (% RPD)	Accuracy (% Rec.)	Precision (% RPD)	MDL	Reporting Limit	En Chem SOP #
Dichlorofluoromethar	ug/L	5030	8260B	N/A	N/A	75-156	0-9	0.51	2.0	VOA-5
Diethyl ether	ug/L	5030	8260B	70-130	0-40	77-142	0-12	0.51	1.0	VOA-5
Ethyl Acetate	ug/L	5030	8260B	N/A	N/A	70-130	0-40	0.75	5.0	VOA-5
Ethyl methacrylate	ug/L	5030	8260B	N/A	N/A	79-125	0-13	0.26	5.0	VOA-5
Ethylbenzene	ug/L	5030	8260B	93-124	0-10	93-127	0-7	0.39	1.0	VOA-5
Ethylene Oxide	ug/L	5030	8260B	N/A	N/A	70-130	0-40	18.5	200.0	VOA-5
Hexachlorobutadiene	ug/L	5030	8260B	70-130	0-40	63-140	0-12	0.41	1.0	VOA-5
Hexachloroethane	ug/L	5030	8260B	N/A	N/A	74-138	0-8	0.46	1.0	VOA-5
Iodomethane	ug/L	5030	8260B	N/A	N/A	52-174	0-14	0.24	5.0	VOA-5
Isobutanol	ug/L	5030	8260B	N/A	N/A	70-130	0-40	14.38	100.0	VOA-5
Isobutyl Acetate	ug/L	5030	8260B	N/A	N/A	70-130	0-40	0.72	5.0	VOA-5
Isopropanol	ug/L	5030	8260B	N/A	N/A	70-130	0-40	1.0	1000.0	VOA-5
Isopropyl Acetate	ug/L	5030	8260B	N/A	N/A	70-130	0-40	0.39	20.0	VOA-5
Isopropyl benzene	ug/L	5030	8260B	70-130	0-40	89-130	0-7	0.24	1.0	VOA-5
Methacrylonitrile	ug/L	5030	8260B	N/A	N/A	70-130	0-40	0.51	5.0	VOA-5
Methyl tert - butyl Eth	ug/L	5030	8260B	70-130	0-40	81-129	0-10	0.38	1.0	VOA-5
Methyl Acetate	ug/L	5030	8260B	N/A	N/A	70-130	0-40	1.37	5.0	VOA-5
Methyl Methacrylate	ug/L	5030	8260B	N/A	N/A	80-126	0-15	0.54	5.0	VOA-5
Methylcyclohexane	ug/L	5030	8260B	N/A	N/A	70-130	0-40	0.33	5.0	VOA-5
Methylene chloride	ug/L	5030	8260B	84-130	0-10	83-136	0-8	0.69	1.0	VOA-5
n - Butanol	ug/L	5030	8260B	N/A	N/A	70-130	0-40	100.0	100.0	VOA-5
n - Butyl Acetate	ug/L	5030	8260B	N/A	N/A	70-130	0-40	1.0	5.0	VOA-5
n - Butylbenzene	ug/L	5030	8260B	70-130	0-40	83-138	0-8	0.32	1.0	VOA-5
n - Hexane	ug/L	5030	8260B	N/A	N/A	70-130	0-40	0.35	5.0	VOA-5
n - Heptane	ug/L	5030	8260B	N/A	N/A	70-130	0-40	0.75	5.0	VOA-5
n - Propanol	ug/L	5030	8260B	N/A	N/A	70-130	0-40	10	10.0	VOA-5
n - Propyl Acetate	ug/L	5030	8260B	N/A	N/A	70-130	0-40	1.0	20.0	VOA-5
n - Propylbenzene	ug/L	5030	8260B	70-130	0-40	90-133	0-6	0.30	1.0	VOA-5
Naphthalene	ug/L	5030	8260B	70-130	0-40	65-138	0-16	0.45	1.0	VOA-5
p - Isopropyltoluene	ug/L	5030	8260B	70-130	0-40	81-136	0-7	0.27	1.0	VOA-5
Propionitrile	ug/L	5030	8260B	N/A	N/A	70-130	0-40	1.24	20.0	VOA-5
sec - Butylbenzene	ug/L	5030	8260B	70-130	0-40	91-134	0-7	0.39	1.0	VOA-5

EN CHEM MADISON LABORATORY ANALYTICAL METHODS, QA OBJECTIVES FOR AQUEOUS SAMPLES

Effective June 2001 to June 2002 5/4/01

Parameter	units	Method (prep)	Method (analytical)	LCS (% Rec.)	LCS (% RPD)	Accuracy (% Rec.)	Precision (% RPD)	MDL	Reporting Limit	En Chem SOP #
Styrene	ug/L	5030	8260B	90-117	0-10	87-125	0-7	0.24	1.0	VOA-5
tert - Butylbenzene	ug/L	5030	8260B	70-130	0-40	91-132	0-6	0.28	1.0	VOA-5
Tetrachloroethene	ug/L	5030	8260B	91-123	0-10	81-134	0-7	0.39	1.0	VOA-5
Tetrahydrofuran	ug/L	5030	8260B	70-130	0-40	59-133	0-20	0.70	5.0	VOA-5
Toluene	ug/L	5030	8260B	91-126	0-10	90-128	0-8	0.42	1.0	VOA-5
trans - 1,2 - Dichloro	ug/L	5030	8260B	79-125	0-10	86-140	0-9	0.38	1.0	VOA-5
trans - 1,3 - Dichloro	ug/L	5030	8260B	80-137	0-10	81-127	0-7	0.39	1.0	VOA-5
trans - 1,4 - Dichloro	ug/L	5030	8260B	N/A	N/A	51-148	0-14	0.62	5.0	VOA-5
Trichloroethene	ug/L	5030	8260B	85-130	0-10	79-134	0-7	0.44	1.0	VOA-5
Trichlorofluorometha	ug/L	5030	8260B	50-150	0-50	88-163	0-8	0.93	2.0	VOA-5
Vinyl acetate	ug/L	5030	8260B	N/A	N/A	48-168	0-14	0.50	1.0	VOA-5
Vinyl chloride	ug/L	5030	8260B	67-125	0-11	82-149	0-13	0.77	2.0	VOA-5
Xylene, total	ug/L	5030	8260B	94-117	0-13	87-129	0-8	0.71	3.0	VOA-5
Xylenes, -m, -p	ug/L	5030	8260B	85-129	0-10	85-130	0-15	0.69	2.0	VOA-5
Xylenes, -o	ug/L	5030	8260B	83-125	0-6	89-127	0-6	0.76	1.0	VOA-5

EN CHEM MADISON LABORATORY ANALYTICAL METHODS, QA OBJECTIVES FOR AQUEOUS SAMPLES

Effective June 2001 to June 2002 5/4/01

Parameter	units	Method (prep)	Method (analytical)	LCS (% Rec.)	LCS (% RPD)	Accuracy (% Rec.)	Precision (% RPD)	MDL	Reporting Limit	En Chem SOP #
Wet Chemistry										
Nitrogen, nitrate + nitrite -	mg/L	N/A	300.0	90-116	N/A	63-132	0-3	0.15	0.20	WCM-60
Nitrogen, nitrate - Ion Chrc	mg/L	N/A	300.0	90-110	N/A	90-110	0-6	0.13	0.20	WCM-60

EN CHEM GREEN BAY LABORATORY ANALYTICAL METHODS, QA OBJECTIVES FOR AQUEOUS SAMPLES											
	Effective June 2001 to June 2002								5/4/01		
Parameter	Units	Method (prep)	Method (analytical)	LCS (% Rec.)	LCS (% RPD)	Accuracy (% Rec.)	Precision (% RPD)	MDL	Reporting Limit	W. Length Alt. Isotope	SOP's
Volatiles											
Methane, Ethane, Ethene, & Propane List											
Ethane	ug/L	5030B	Headspace	70-130	0-20	70-130	0-20	1.6	5.6	N/A	G3-VOA-18
Ethene	ug/L	5030B	Headspace	70-130	0-20	70-130	0-20	1.4	5.0	N/A	G3-VOA-18
Methane	ug/L	5030B	Headspace	70-130	0-20	70-130	0-20	2.0	2.8	N/A	G3-VOA-18
Propane	ug/L	5030B	Headspace	70-130	0-20	70-130	0-20	3.2	8.0	N/A	G3-VOA-18
					contro., lcs		sure				

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	
Wet Chemistry											
Total organ	mg/kg	9060	9060	86-130	N/A	35-155	0-16	91	500	N/A	WCM-9

EN CHEM MADISON LABORATORY ANALYTICAL METHODS, QA OBJECTIVES FOR NON-AQUEOUS SAMPLES											
		Effective June 2001 to June 2002							5/4/01		
Parameter	Units	Method (prep)	Method (analytical)	LCS (% Rec.)	LCS (% RPD)	Accuracy (% Rec.)	Precision (% RPD)	MDL	Reporting Limit	W. Length Alt. Isotope	En Chem SOP #
Wet Chemistry											
Total organ	mg/kg	9060	9060	86-130	N/A	35-155	0-16	91	500	N/A	WCM-9

EN CHEM MADISON LABORATORY ANALYTICAL METHODS, QA OBJECTIVES FOR NON-AQUEOUS SAMPLES											
		Effective June 2001 to June 2002							5/4/01		
Parameter	Units	Method (prep)	Method (analytical)	LCS (% Rec.)	LCS (% RPD)	Accuracy (% Rec.)	Precision (% RPD)	MDL	Reporting Limit	W. Length Alt. Isotope	En Chem SOP #
Volatiles											
Target Compound List - 4.2 (w/ Method 8260B)											
1,1 - Dichlo	ug/kg	5035	8260B	70-130	0-40	43-156	0-26	1.05	5.0	N/A	VOA-9
1,1 - Dichlo	ug/kg	5035	8260B	53-138	0-10	36-169	0-24	1.24	5.0	N/A	VOA-9
1,1,1 - Tric	ug/kg	5035	8260B	70-130	0-40	50-144	0-26	1.03	5.0	N/A	VOA-9
1,1,2 - Tric	ug/kg	5035	8260B	70-130	0-40	61-126	0-21	1.32	10.0	N/A	VOA-9
1,1,2 - Tric	ug/kg	5035	8260B	70-130	0-40	67-127	0-13	1.22	5.0	N/A	VOA-9
1,1,2,2 - Te	ug/kg	5035	8260B	70-130	0-40	D-209	0-27	0.83	5.0	N/A	VOA-9
1,2 - Dibro	ug/kg	5035	8260B	70-130	0-40	6-162	0-29	1.11	5.0	N/A	VOA-9
1,2 - Dibro	ug/kg	5035	8260B	70-130	0-40	57-128	0-13	0.94	5.0	N/A	VOA-9
1,2 - Dichlo	ug/kg	5035	8260B	70-130	0-40	36-130	0-21	0.92	5.0	N/A	VOA-9
1,2 - Dichlo	ug/kg	5035	8260B	70-130	0-40	50-143	0-11	1.21	5.0	N/A	VOA-9
1,2 - Dichlo	ug/kg	5035	8260B	70-130	0-40	55-137	0-8	0.87	5.0	N/A	VOA-9
1,2,4 - Tric	ug/kg	5035	8260B	70-130	0-40	D-135	0-19	1.05	5.0	N/A	VOA-9
1,3 - Dichlo	ug/kg	5035	8260B	70-130	0-40	34-134	0-22	0.79	5.0	N/A	VOA-9
1,4 - Dichlo	ug/kg	5035	8260B	70-130	0-40	29-135	0-25	1.24	5.0	N/A	VOA-9
2 - Butano	ug/kg	5035	8260B	70-130	0-40	70-130	0-40	1.56	10.0	N/A	VOA-9
2 - Hexano	ug/kg	5035	8260B	70-130	0-40	70-130	0-40	1.93	10.0	N/A	VOA-9
4 - Methyl -	ug/kg	5035	8260B	70-130	0-40	70-130	0-40	1.79	10.0	N/A	VOA-9
Acetone	ug/kg	5035	8260B	70-130	0-40	70-130	0-40	5.54	10.0	N/A	VOA-9
Benzene	ug/kg	5035	8260B	74-133	0-8	43-151	0-18	0.94	5.0	N/A	VOA-9
Bromodich	ug/kg	5035	8260B	70-130	0-40	57-124	0-13	0.95	5.0	N/A	VOA-9
Bromoform	ug/kg	5035	8260B	70-130	0-40	51-115	0-13	0.85	5.0	N/A	VOA-9
Bromometh	ug/kg	5035	8260B	50-150	0-50	63-169	0-24	1.14	10.0	N/A	VOA-9
Carbon dis	ug/kg	5035	8260B	70-130	0-40	70-130	0-40	1.18	5.0	N/A	VOA-9
Carbon tet	ug/kg	5035	8260B	70-130	0-40	43-143	0-30	1.21	5.0	N/A	VOA-9
Chlorobenz	ug/kg	5035	8260B	86-129	0-7	66-114	0-20	0.77	5.0	N/A	VOA-9

EN CHEM MADISON LABORATORY ANALYTICAL METHODS, QA OBJECTIVES FOR NON-AQUEOUS SAMPLES											
		Effective June 2001 to June 2002							5/4/01		
Parameter	Units	Method (prep)	Method (analytical)	LCS (% Rec.)	LCS (% RPD)	Accuracy (% Rec.)	Precision (% RPD)	MDL	Reporting Limit	W. Length Alt. Isotope	En Chem SOP #
Chlorodibr	ug/kg	5035	8260B	70-130	0-40	44-133	0-27	0.84	5.0	N/A	VOA-9
Chloroetha	ug/kg	5035	8260B	50-150	0-50	50-161	0-18	1.97	10.0	N/A	VOA-9
Chloroform	ug/kg	5035	8260B	70-130	0-40	50-145	0-16	1.07	5.0	N/A	VOA-9
Chlorometh	ug/kg	5035	8260B	50-150	0-50	51-167	0-22	1.2	10.0	N/A	VOA-9
cis - 1,2 - D	ug/kg	5035	8260B	70-130	0-40	64-126	0-15	1.10	5.0	N/A	VOA-9
cis - 1,3 - D	ug/kg	5035	8260B	70-130	0-40	58-122	0-14	1.14	5.0	N/A	VOA-9
Cyclohexan	ug/kg	5035	8260B	70-130	0-40	70-130	0-40	1.10	10	N/A	VOA-9
Dichlorodif	ug/kg	5035	8260B	50-150	0-50	33-162	0-22	2.57	10	N/A	VOA-9
Ethylbenze	ug/kg	5035	8260B	70-130	0-40	58-129	0-27	2.15	5.0	N/A	VOA-9
Isopropyl b	ug/kg	5035	8260B	70-130	0-40	49-134	0-20	1.01	5.0	N/A	VOA-9
Methyl - ter	ug/kg	5035	8260B	70-130	0-40	70-130	0-40	0.96	5.0	N/A	VOA-9
Methyl Ace	ug/kg	5035	8260B	70-130	0-40	70-130	0-40	1.32	10	N/A	VOA-9
Methylcycl	ug/kg	5035	8260B	70-130	0-40	70-130	0-40	1.16	10	N/A	VOA-9
Methylene	ug/kg	5035	8260B	70-130	0-40	40-154	0-17	0.99	5.0	N/A	VOA-9
Styrene	ug/kg	5035	8260B	70-130	0-40	D-163	0-50	0.97	5.0	N/A	VOA-9
Tetrachlor	ug/kg	5035	8260B	70-130	0-40	58-137	0-26	1.14	5.0	N/A	VOA-9
Toluene	ug/kg	5035	8260B	85-131	0-8	65-128	0-17	1.28	5.0	N/A	VOA-9
trans - 1,2	ug/kg	5035	8260B	70-130	0-40	64-132	0-26	1.11	5.0	N/A	VOA-9
trans - 1,3	ug/kg	5035	8260B	70-130	0-40	55-125	0-16	1.07	5.0	N/A	VOA-9
Trichloroet	ug/kg	5035	8260B	60-132	0-10	22-179	0-22	0.93	5.0	N/A	VOA-9
Trichloroflu	ug/kg	5035	8260B	50-150	0-50	55-148	0-22	1.56	10	N/A	VOA-9
Vinyl chlori	ug/kg	5035	8260B	50-150	0-50	42-162	0-20	1.89	10.0	N/A	VOA-9
Xylene, tot	ug/kg	5035	8260B	70-130	0-40	54-131	0-22	2.69	15.0	N/A	VOA-9

APPENDIX E.

ENCHEM INC. QUALITY ASSURANCE PLAN

QUALITY MANUAL
FOR
EN CHEM, INC. – GREEN BAY, WI

REVISION 0

Prepared by:
En Chem, Inc.
1795 Industrial Drive, Green Bay, WI 54302
Ph: (920)-469-2436, Fax: (920)-469-8827
www.encheminc.com

EFFECTIVE DATE:
March 01 , 2001

David Turniff, Laboratory Director — Date: 02/26/2001

Michael C. Suha, QA Officer — Date: 02/26/2001

Jeff Bushner, Technical Director — Date: 2/26/2001

Nickolas Severin, Technical Director — Date: 02/26/2001

Nils Melberg, Operations Manager — Date: 2/26/2001

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1.0 POLICY STATEMENT

This Quality Manual summarizes the policies and operational procedures associated with En Chem, Inc. (En Chem) laboratory located in Green Bay, WI. Specific protocols for sample handling and storage, chain-of-custody, laboratory analyses, data reduction, corrective action, and reporting are described. Adhereance to procedures listed in this manual shall be the responsibility of all En Chem employees of the Green Bay 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.

The En Chem Green Bay Laboratory performs chemical analyses for inorganic and organic constituents in aqueous and solid 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 Green Bay. 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.

1.1 Mission Statement

It is En Chem's mission to pro-actively serve our customers by continually improving the cost effectiveness and quality of our products and services. We will accomplish this by building a team of professionals who develop and maintain a quality and service oriented attitude that leads the industry and creates a sustainable advantage for En Chem.

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 which 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.

2.0 ORGANIZATION AND RESPONSIBILITIES

2.1 Organization Chart

An organizational chart for En Chem is shown in Appendix C.

A listing of credentials for all laboratory staff is presented in Appendix D.

2.2 Laboratory 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 the Technical Director or 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 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. The QA Officer 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.

2.5 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.

2.6. 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 G1-LAB-8 document the use of current SOPs. Analysts and Technicians demonstrate continued proficiency by 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.

2.7 Laboratory Capabilities

The En Chem Green Bay laboratory performs analyses on aqueous and solid 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 Green Bay 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:

$$\text{RPD} = \frac{[D_1 - D_2]}{\left[\frac{(D_1 + D_2)}{2}\right]} \times 100$$

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 sample.
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 to be obtained 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 Detection Limits

3.6.1 Method Detection Limits

Method Detection Limits (MDLs) are determined for all analytes as specified in the laboratory SOP G1-QUA-2. The laboratory SOP is based on EPA guidance given in Title 40, Code of Federal Regulations, Part 136, Appendix B.

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.

4.0 SAMPLING PROCEDURES

The 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

Sample containers are normally provided by the laboratory. 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 G1-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 G1-LAB-09, Traceability of Laboratory Reagents.

Proper preservation is the ultimate responsibility of the sampling team. Additional preservatives may be supplied by the laboratory for any site with a history indicating that non-routine preservation is required. Proper sample preservation is verified by the laboratory at the time of sample receipt and adjustments with proper documentation are made as necessary.

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, will 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

Sample collection, preservation, and storage, before analysis must be performed properly to maintain sample integrity. En Chem's laboratories are access-controlled facilities. Upon receipt all samples are 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. 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 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 ending with delivery to the laboratory. 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 serves 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 initialed and dated at the time that they are made. *If the discrepancies cannot be resolved, project personnel will be notified and the samples in question 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 Chain of Custody supplied with the samples and is completed for each batch of samples received. The use of this laboratory form is discussed in SOP G1-REC-8.

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 and sample log-in (SOP G1-REC-7). Refer to the specific laboratory SOP for further detail.

5.3.1 Sample Acceptance Policy

En Chem's written sample acceptance policy 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 are documented on a Sample Entry Nonconformance Memo (SOP G1-REC-5). 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 must 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 properly stored.

To minimize the possibility of contamination all samples for volatile organics are segregated in a refrigerator specifically designated for these samples. A second refrigerator is used to segregate known high level samples, or those with a noticable odor at the time of receipt.

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 Green Bay 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 as required for the open container policy. Hazardous wastes are shipped to licensed waste disposal facilities for disposal. En Chem receives a Certificate of Disposal for all disposed material. These documents are maintained on file in the laboratory.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

Instruments and equipment used to generate data are 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

Calibration of analytical support equipment and instruments is traceable to the National Institute of Standards and Technology (NIST).

6.2 Reference Standards

Physical standards (e.g., weights, thermometers) are traceable to nationally recognized standards such as the NIST, which are at least four to ten times as accurate as the equipment requirements. Physical standards are verified annually, at a minimum, by a certified external agency.

Chemical reference standards are purchased from commercial vendors and are traceable to the NIST. Certificates which accompany standard materials when received in the laboratory are maintained on file in the laboratory.

6.3 General Requirements

Each calibration is supported by documentation indicating calibration date, method, instrument, analysis date, analyte, concentration and response (or response factor). Sufficient information is 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.

At a mimimum the following information is recorded during verification:

- NIST Reference serial number used for comparison (eg. Thermometer or Class S mass)
- Measurement of NIST Reference
- En Chem identification of laboratory equipment
- Measurement of En Chem laboratory equipment
- Date of verification
- Personnel performing the verification

NOTE: Separate records are kept for periodic calibration. These items are 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 are kept on file.

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.

6.4.1 Temperature Monitoring

Each working day, the temperatures of ovens, refrigerators, and freezers 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 G1-LAB-12 and G1-LAB-13 describe these procedures and corrective action for noncompliance.

All therometers used in the laboratory are verified 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-10.

6.4.2 Balances

Laboratory balances are checked with Class 'S' weights traceable to NIST standards over the entire range of use each day prior to use. The calibration check is recorded in the logbook which contains the acceptance limits for that unit. SOP G1-REC-1 describes this procedure and corrective action.

All balances are serviced and calibrated atleast 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 are maintained by the QA Officer.

6.4.3 Volumetric Dispensing Devices

Mechanical volumetric dispensing devices used where the volume dispensed affects calculated results such as adjustable pipettes and re-pipettors are checked for accuracy monthly as described in laboratory SOP G1-LAB-1A.

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 is 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 is recorded or referenced in the logbook: instrument identification, calibration date, analyst, calibration solutions/concentrations analyzed.

6.5.1 Limited Calibration Procedures

Under a project specific basis and where certification does not exist, the use of a limited calibration procedure is allowed 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, or establish a reporting limit. The use of limited calibration will be discussed with the client by the Project Manager prior to initiation of the project. The agreement to proceed with a limited calibration for a project will be documented in the project file by the Project Manager. The results derived from such a procedure are qualified as to the origin.

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).

Deviations from established procedures are documented by the use of a nonconformance memo. See Section 13. The memo is then used to produce comments or a formal narrative sent with the final report to the client.

7.1 SOP Preparation and Organization

Laboratory SOP G1-LAB-1, Preparation of Standard Operating Procedures, defines the format, identification, and control of SOPs.

7.2 SOP Control and Distribution

SOPs are located throughout the laboratory accessed by an icon on computer desktops and are printed on paper with a red header signifying that the copy is a controlled document. The controlled copies are issued and distributed by the QA Officer or a designee. Each laboratory area has elcetronic access to copies of the appropriate SOPs pertaining to that work area. The SOPs are accessible to all personnel in their immediate work areas.

SOPs may be sent to clients for inclusion in workplans, etc. These copies are considered uncontrolled and are photocopies of the controlled SOPs. They do not have the red header and are easily identified as uncontrolled copies.

7.3 SOP Archival

When an SOP is revised, the original of the previous revision is archived by the QA Officer. These archived SOPs are retained indefinitely for future reference. The archive is maintained in a file cabinet for safe keeping.

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 Green Bay SOPs is located in Appendix J.

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 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. For authoritative source methods without defined QC requirements En Chem has adopted a policy from Standard Methods and is outlined in SOP G1-LAB-2.

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. Routinely, Trip Blanks are prepared by filling two 40-mL HCl preserved VOA vials with organic free water. Where appropriate two 40-mL unpreserved VOA vials with organic free water shall be sent. 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 blanks and duplicates are used as controls.

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 <u>all</u> 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 75-125. 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 for example 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.

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 the analysis of QC check samples or calibration standards. The detection limit for gravimetric tests is based on

a) the analysis of seven replicates of a purchased QC sample. Alternatively, it 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.

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. This would be discussed with Laboratory Project Management Staff to reach agreement of the deviations from standard reporting limits prior to sample shipment.

MDLs are updated when major changes are made to the processing of samples by use of an update procedure, but are assessed no less than once annually.

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 would be performed to verify a compound identification when positive results are detected and questionable for GC analysis. Such confirmations are performed on organic tests except when the analysis involves the use of a mass spectrometer or methods involving the quantitation of a class of compound (e.g. - TPH - Diesel).

8.4 Demonstration of Method Capability

Prior to acceptance and use of any method, satisfactory initial demonstration of method performance is required. This initial demonstration of method performance is performed each time there is a significant change in instrument type, personnel or test method. 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 Analytical SOPs document 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 typically 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 as comments or formal narrative, and 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 all test data provided by outside sources, such as subcontracted laboratories, clients, etc.
11) clear identification of numerical results with values below the Reporting Limit.
12) a signature of laboratory personnel accepting responsibility for the content of the report
13) date of issuance of the report.

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 Formats

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.

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. 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 G1-DOC-03.

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 G1-LAB-08.

All quality documents (this Manual, SOPs, policies, etc.) are reviewed and approved by the QA Officer, the Technical Directors and the Laboratory Director. 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 1-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 which is sent to, or received from, clients will be maintained in the project file for that project 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 G1-QAU-5.

10.2 Performance Audits

Proficiency test samples are analyzed four 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. These are single blinds. At times at the discretion of the QA Officer a double blind maybe more suitable and is purchased.

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 this 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 department in the laboratory, i.e. metals, semivolatile organics, volatile organics. 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 G1- LAB-04.

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 which 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 G1-LAB-03.

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:

- Section 8.1 through 8.11 of the EPA Document "2185 - Good Automated Laboratory Practices" (1995), is used as the standard.
- 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.

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 is covered under manufacturer service contacts. Periodic preventive maintenance is performed by manufacturer service technicians or factory trained En Chem staff. Daily or routine preventive maintenance is performed by the analyst responsible for the instrument. 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 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 are to 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 taken from 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	Accuracy Targets % Recovery	Precison Targets %RPD
Metals	75-125	20
Volatile Organics	70-130	40
Volatile Gases	50-150	50
Base/neutrals	70-130	40
Acids	40-140	40

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. En Chem uses an "A" for Inorganic analysis and "B" for Organic analysis to qualify data in regards to the method blank. In both cases the the level of analyte present in the method blank is provided.

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 which can affect data quality. All out-of-control situations or deviations from SOP are documented on a nonconformance memo. All En Chem employees are responsible for initiating a nonconformance memo for any situation which deviates from laboratory practice or SOP. Laboratory SOP G1-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 procedures for resolution of client complaints is documented in SOP G1-GEN-29.

14.0 SUBCONTRACTING AND SUPPORT SERVICES AND SUPPLIES

14.1 Subcontracting Laboratory Services

En Chem clients are 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 G1-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 1999

Example NELAC Quality Manual, Catalyst Information Resources, L.L.C., Evergreen, CO

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

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.
1795 Industrial Drive
Green Bay, WI 54302

Signature	David Turiff Laboratory Director/ President	02/26/2001 Date
Signature	Michael C. Suha Quality Assurance Officer	02/26/01 Date
Signature	Jeffery J. Bushner Technical Director	2/26/2001 Date
Signature	Nickolas J. Severin Technical Director	02/26/01 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) of known concentration 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 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.
1795 Industrial Drive
Green Bay, WI 54302

Analyst(s) Name(s)_____

Matrix: Reagent Water____ Ottowa Sand _____

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.

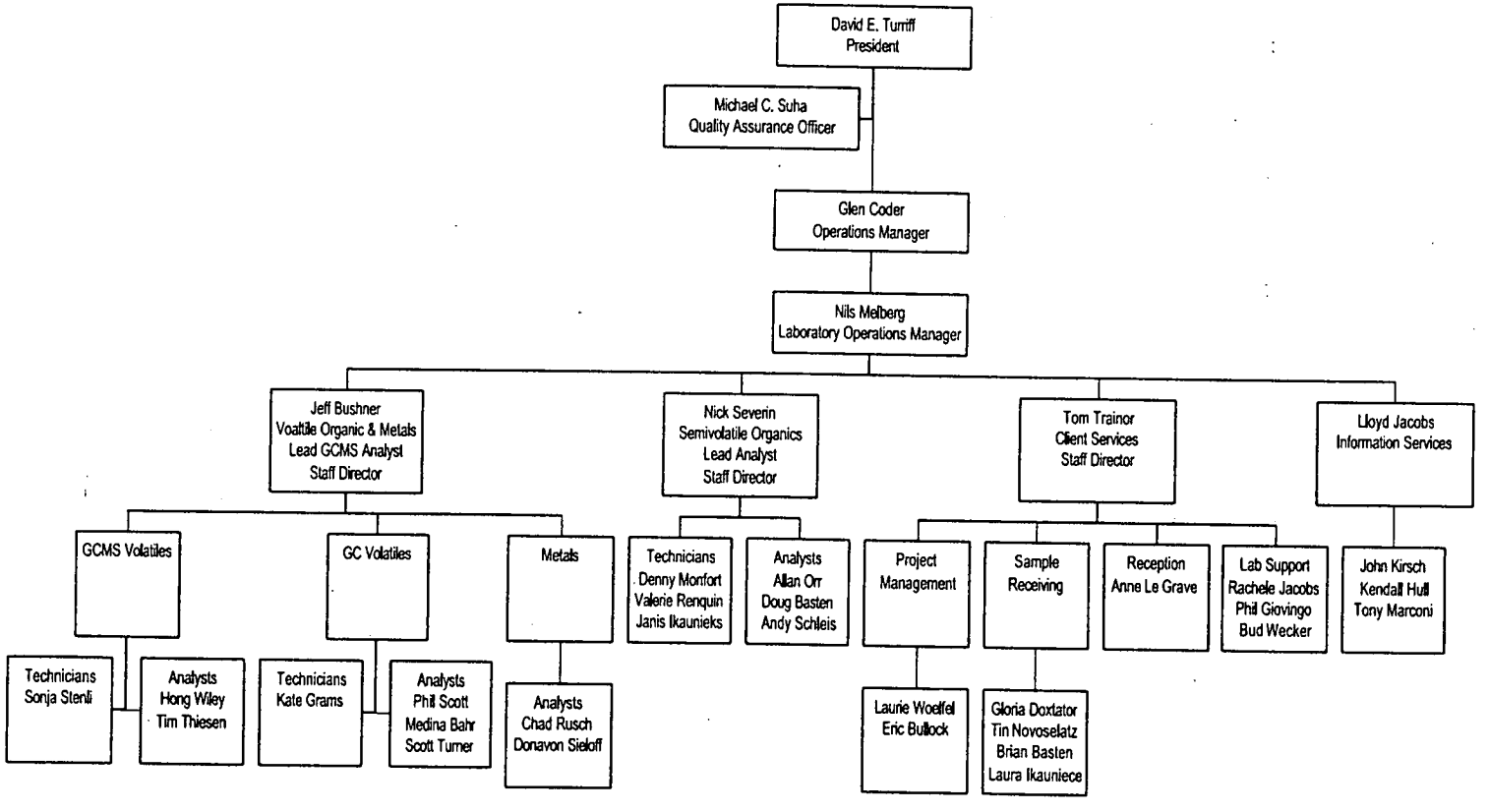
5. All raw data(including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained, and, the associated information is well organized and available for review by authorized inspectors.

_____ _____
Laboratory Manager Signature/Date

_____ _____
Quality Assurance Officer Signature/Date

APPENDIX C

ORGANIZATIONAL CHART



Green Bay Facility – January 2001

APPENDIX D

PERSONNEL SUMMARY

APPENDIX D: EMPLOYEE CREDENTIALS

NAME	TITLE	DEGREE	YEARS EXP.
Nils K. Melberg	Operations Manager	B.S. Water Chemistry	14
Project Management			
Thomas J. Trainor	Lead Project Manager	B.S. Chemical Engineering	13
Laurie K. Woelfel	Project Manager	H.S.	13
Eric Bullock	Project Manager	B.S. Environmental Sciences	3
Project Coordinators			
Brian Basten	Project Coordinator	H.S.	5
Information Systems			
Lloyd Jacobs	MIS Technician	H.S.	27
John Kirsch	MIS Technician	A.S. Microcomputers	3
Kendall Hull	MIS Technician	M.S. Paper Science	2
Administrative Support			
Judy Theys	Purchasing Agent	B.S. Chemistry	12
Anne Le Grave	Administrative Assistant	H.S.	3
Ron Sommerhaulder	Human Resources	B.S. Mathematics	5
Inorganic Chemistry - Metals			
Chad R. Rusch	Group Leader	B.S. Chemistry	4
Donovan Seilloff	Senior Analyst	B.S. Chemistry	2
Organic Chemistry - Semivolatile			
Nickolas J. Severin	Technical Director Group Leader	B.S. Chemistry	7
Alan Orr	Laboratory Analyst	B.S. Biology	7
Douglas J. Basten	Laboratory Analyst	B.S. Biology	7
Andrew Schleis	Laboratory Analyst	B.S. Chemistry	2
Denny Monfort	Laboratory Technician	B.S. Biology	2
Valerie Renquin	Laboratory Technician	H.S.	4
Janis Ikaunieks	Laboratory Technician	M.S. Chemical Engineering	3

Organic Chemistry - Volatile GC/MS			
Jeffrey J. Bushner	Technical Director Senior Analyst	B.S. Geology	15
Randy Naidl	Laboratory Analyst	B.S. Chemistry	8
Hong Wiley	Laboratory Analyst	B.S. Chemistry	9
Tim Thiesen	Laboratory Analyst	B.S. Geology	13
Sonja Stenli	Laboratory Technician	B.S. Environmental Sciences	2
Organic Chemistry - Volatile GC			
Phil Scott	Senior Analyst	B.S. Water Resources Chemistry	14
Medina Bahr	Laboratory Analyst	B.S. Biology	7
Scott Turner	Laboratory Analyst	B.S. Water Chemistry	5
Kate Grams	Laboratory Technician	B.S. Soil Science	2
Sample Receiving			
Tim Novoselatz	Group Leader	B.S. Environmental Science	6
Gloria Doxtator	Laboratory Technician	H.S.	12
Phil Giovingo	Laboratory Technician	H.S.	4
Laura Ikauniece	Laboratory Technician	M.S. Environmental Engineering	3
Quality Assurance			
M. Suha	QA Officer	B.S. Zoology, CHMM	11
Jill Duranceau	QA Auditor	B.S. Forestry	12

+ 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					
		SM/ EPA-600	SW-846	SOP	
Matrix					
pH	pH Probe	150.1	9040B/9045C	G2-WCM-3	W,S
Solids, total	Gravimetric	SM2450		G2-WCM-4	S
Solids, total suspended	Gravimetric	160.2	—	G2-WCM-2	W
Turbidity		180.1	—	G2-MET-11	W
Ignitability	Pensky-Martin	—	1010	G2-WCM-1	W,S
Hardness by Calculation	ICP/MS	SM2340B	—	G2-MET-16	W

EPA-600	EPA-600/4-79-020
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-846	
Acid Digestion - ICP or ICPMS - Dissolved or Total Recov. Metals	—	3005A	G2-MET-00
Acid Digestion of Aqueous Samples for Total Metals Analysis by ICPMS	—	3020	G2-MET-02

COMPOSITIONAL

	EPA-600	SW-846	
Acid Digestion - ICPMS	—	3050B	G2-MET-12
Acid Digestion - ICPMS -Ag, Sb	—	3050B [2]	G2-MET-13

2: Includes use of option 7.5 of the method.

Metals Analysis

	EPA-600	SW-846	
Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy	200.8	6020	G2-MET-14

Other Capabilities

None

APPENDIX E
LABORATORY CAPABILITIES

METHOD REFERENCE EN CHEM SOP

Volatiles Preparation

	EPA-600	SW-846	
Volatile Organic Preparation of Solid Matrices	----	5035	G3-VOA-20
Volatile Organic Preparation of Aqueous Matrices and High Concentration Soils	----	5030B	G3-VOA-03

Volatiles Analysis

	EPA-600	SW-846	
Volatile GC/MS Analysis	----	8260B	G3-VOA-01
Aromatic And Halogenated Volatiles By Gas Chromatography Using Photoionization Detectors	----	8021B	G3-VOA-19
Analysis of Dissolved Methane, Ethane, Propane, and Ethylene in Ground Water	---- Headspace		G3-VOA-18
Total Petroleum Hydrocarbons - Gas	----	8015B	G3-GRO-05
Iowa Gasoline Range Organics	----	OA-1	G3-GRO-03
Wisconsin Modified Gasoline Range Organics	----	WDNR-95	G3-GRO-02

Other Capabilities

None

APPENDIX E
LABORATORY CAPABILITIES

METHOD REFERENCE EN CHEM SOP

SemiVolatile Extractions

	EPA-600	SW-846	
Aqueous Sample Preparation for the Analysis of Base/Neutral/Acids Full Scan	USEPA 40CFR 625	3510C	G3-SVO-20
Aqueous Sample Preparation for the Analysis of Polynuclear Aromatic Hydrocarbons by HPLC	USEPA 40CFR 610	3510C	G3-SVO-08
Soil/Semisolid Sample Preparation for the Analysis of Semivolatile Organics in Soil.	—	3545	G3-SVO-09
Soil/Semisolid Sample Preparation for the Analysis of Semivolatile Base/Neutral/Acids - Full scan	USEPA 40CFR 625	3545	G3-SVO-21

SemiVolatile Analysis

	EPA-600	SW-846	
WI Modified DRO	—	WDNR 95	G3-SVO-01
HPLC Analysis of Polynuclear Aromatic Hydrocarbons	610	8310	G3-SVO-02
Total Petroleum Hydrocarbons - GC	—	8015B	G3-SVO-03
Analysis of Polynuclear Aromatic Hydrocar by GC/MS - SIMs Mode	—	8270C	G3-SVO-04
Iowa Diesel Range Organics	—	OA-2	G3-SVO-05
TNRCC C6 To C28 Petroleum Hydrocarbons	—	TNRCC	G3-SVO-06
Petroleum Range Organics - Florida	—	FLPRO	G3-SVO-11
Tennessee Extractable Petroleum Hydrocarbons	—	TN-EX	G3-SVO-12
Analysis of PCB Arochlors by GC	608	8082	G3-SVO-18
Analysis of Base/Neutral and Acid (BNA) Compounds by GC/MS - Full Scan	625	8270C	G3-SVO-19

Other Capabilities

CLEANUP

	EPA-600	SW-846	
Sulfuric Acid Cleanup	—	3665A	G3-SVO-17

APPENDIX F

LABORATORY INSTRUMENT INVENTORY

INSTRUMENT INVENTORY	
Section **Instrument/Peripherals**	**Date of Purchase**
METALS	
1. ICP-MS , Hewlett Packard 4500 Series	2000
SEMIVOLATILES	
1. TPH - Extractables a. Hewlett Packard GC/dual FID 5890II - 4 instruments	1992-1996
2. Aqueous Poly Aromatic Hydrocarbons a.HPLC - Hewlett Packard - 1 instrument 1046A Fluorescence Detector + DAD	1994
3. Poly Aromatic Hydrocarbons a. Hewlett Packard GC/MS 6890 - 1 instrument 5972 Mass Spec Detector	1996
b. Hewlett Packard GC/MS 6890 5973 Mass Spec Detector	2000
4. Polychlorinated Biphenyl a. Hewlett Packard 5890II - 1 instrument Single ECD Detector with TDX EZ Flash	2000
5. Base Nuetral and Acids Hewlett Packard GC/MS 5890 - 2 instruments 5972 Mass Spec Detector	2000
VOLATILES - GC	
1. TPH - Purgeable and Aromatic Volatiles a.Hewlett Packard GC/PID-FID 5890II - 2 instruments DynaTech PTA-30 Purge and Trap	1992
b. Hewlett Packard GC/PID-FID 5890II - 3 instruments DynaTech/DynaTrap Autosampler	1992-1996
c. Hewlett Packard GC/PID-FID 5890II - 2 instruments Archon Autosampler	1996-1998
2. a. Hewlett Packard GC 5890II - 2 instruments Headspace with FID	1996-1998
VOLATILES - GCMS	
1. Purgeable Volatiles a.Hewlett Packard GC/ 5890II - 2 instruments Tekmar 3000 Purge and Trap Concentrator 5972 Mass Spec Detector Archon Autosampler	1992-1999
b.Hewlett Packard GC/ 5890II - 3 instruments Tekmar 3000 Purge and Trap Concentrator 5972 Mass Spec Detector DynaTech PTA-30 Purge and Trap	1992-1999

INSTRUMENT INVENTORY	
Section **Instrument/Peripherals**	**Date of Purchase**
WET CHEMISTRY	
1. Precision Scientic - Flashpoint Instrument	1996
2. Orion 230A - pH Meter	1994
SAMPLE PREP/OTHER	
1. Top Loading Balances 5 Units a.Mettler/Toledo	Various
2. Top Loading Balance 1 Unit a. Satorius	1996
1. Analytical Balances - 1 Unit a.Ohaus	Various
1. Drying Ovens - 3 Units a. Fisher Scientific	Various
1. Hot Block Digestion Systems - 2 Units	1999 and 2000
1. Mass Calibrations - 3 Class S Sets a. Tromener	Various

APPENDIX G

LABORATORY FLOORPLAN

Not available in this revision

APPENDIX H

LABORATORY CERTIFICATIONS/APPROVALS

Certification programs vary by Accrediting Authority. Some Authorities do not require laboratory accreditation. The facility participates in many accreditation programs. A summary is given in this appendix, and is intended to be a starting point for discussions of detailed items for a project scope. These discussions are to begin with Sales and Project Management staff members of En Chem.

STATE/ AGENCY	CERTIFICATION NUMBER
Arkansas	—
Florida	CQapp 990073a
Illinois/NELAC	100431
Iowa	135
Louisiana	AI85162
Minnesota	055-999-334
North Dakota	R-150
Oklahoma	9918
South Carolina	83006
Wisconsin	405132750

APPENDIX I

LABORATORY FORMS

Form

Bottle Request Form

Chain of Custody Record

Sample Entry Non Conformance Memo

Preparation/Analysis Non Conformance Report

Bottle Request Form

Madison Office/Laboratory
En Chem, Inc.
525 Science Dr.
Madison, WI 53711
608-232-3300

Green Bay Office/Laboratory
En Chem, Inc.
1241 Bellevue St., Suite 9
Green Bay, WI 54302
920-469-2436

PLEASE SHIP SAMPLES TO: ___**Madison Laboratory** ___ **Green Bay Laboratory**

Client Name:_____
Client Contact:_____
Project #:_____
Project Name:_____
Project State:_____
Sample Date:_____
Sample Type:_____

Bottle Requestor:_____
Date Bottles Needed:_____
Date Requested:_____

Bottle Preparer:_____
Date Prepared:_____
Checked By:_____

Deliver to:_____
_____
_____
_____
Attn:_____
Phone:_____

Shipping Charge:_____
Method of Shipment:_____
Client Fed Ex/UPS #:_____
Cooler #'s: _____
Number/Size of Coolers:_____

EnChem Project Manager/Phone #: _____

Check if Required: __Temp. Blank __Chains of Custody __Labels __Custody Seals __Trip Blank

# of Smpls	# of Bottles	Size	Type	Preserv.	Associated Parameters	Bottle Lot Number

Comments:

(Please Print Legibly)

Company Name: _____

Branch or Location: _____

Project Contact: _____

Telephone: _____

Project Number: _____

Project Name: _____

Project State: _____

Sampled By (Print): _____

EnChem Inc.

☐ 1241 DePere St., Suite 9
Green Bay, WI 54302
920-469-2436
FAX 920-469-4627

☐ 638 Science Drive
Madison, WI 53711
608-233-3300
FAX: 608 233-0502

CHAIN OF CUSTODY

*Preservation Codes
A=None B=HCL C=H2SO4 D=HNO3 E=EnCore F=Methanol G=NaOH
H = Sodium Bisulfate Solution I = Other

FILTERED? (YES/NO)

PRESERVATION (CODE)*

ANALYSES REQUESTED

TOTAL # OF BOTTLES SENT

Page ____ of ____

P.O. # ____ Quote # ____

Mail Report To: _____

Company: _____

Address: _____

Invoice To: _____

Company: _____

Address: _____

Mail Invoice To: _____

Data Package Options
(please circle if requested)
Results Only
EnChem Level III (Subject to Surcharge)
EnChem Level IV (Subject to Surcharge)

Regulatory Programs
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										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: _____

Samples on HOLD are subject to special pricing 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:

EnChem Project No.

Sample Receipt Temp.

Sample Receipt pH

Cooler Custody Seal

Present / Not Present

Intact / Not Intact

Version 1.0-402

SAMPLE ENTRY NONCONFORMANCE MEMO			CLIENT NAME/CONTACT:
EnCHEM PROJECT NO:			PROJECT NAME: PROJECT NUMBER:
Problem	Lab ID	Field ID	Comments/Corrective Action
No Ice			
Broken Bottles/ Cracked Cap			
Hold Time			
Headspace			
Labeling			
Volume			
Preservation			
Container			
Other			

NONCONFORMANCE REPORT

DATE:_____ BLANK NUMBER:_____
(QA ASSIGNED)

LABORATORY

☐ GRO ☐ SVOC ☐ VOC ☐ METALS

INSTRUMENT: ☐ GC ☐ GC/MS ☐ ICPMS ☐ GFAA

NUMBER : ☐ 1 ☐ 2 ☐ 3 ☐ 4 ☐ 5 ☐ 6 INITATOR:_____

☐ BLANK ☐ EXTRACTION BLANK ☐ MATRIX SPIKE/DUPLICATE ☐ LCS/DUPLICATE ☐ CVS

DESCRIBE THE NONCONFORMANCE \ OCCURRENCE_____

_____

_____

_____

_____

CORRECTIVE ACTION TAKEN FOR THIS OCCURRENCE _____

_____

_____

IS THIS DATA BEING ACCEPTED ? ☐ YES ☐ NO

IF YES WHAT IS THE REASON FOR ACCEPTANCE_____

_____

_____

_____

THE DATA IS QUALIFIED WITH ☐ NO QUALIFICATIONS

☐ _____

_____

_____

_____

GROUP LEADERS APPROVAL:_____ DATE:_____

QA MANAGER'S APPROVAL:_____ DATE:_____

CORRECTIVE ACTION FOLLOW-UP COMMENTS_____

_____

COPY ROUTING

☐ GROUP LEADER ☐ PROJECT MANAGER ☐ LAB MANAGER ☐ LAB DIRECTOR

f:\sop\forms\ncmemo.doc THE ORIGINAL WILL BE ON FILE IN THE DAILY DATA PACKAGE

APPENDIX J

SOP LISTING

SOP #	Title	Control		
	Volatile Organics (VOA)	Rev. #	Date	Status
G3-VOA-1	Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry (GC/MS)	4	1/01	FINAL
G3-VOA-2	Soil/Semisolid Sample Preparation for the Analysis of Volatile Organics in soil preserved with Methanol	5	1/01	FINAL
G3-VOA-3	Volatile Organic Preparation of Aqueous Matrices and High Concentration Soils by Method 5030B	3	1/01	FINAL
G3-VOA-4	Cleaning of syringes used in the Analysis of Volatile Organics	4	1/01	FINAL
G3-VOA-5	Extruding Sample from 25g En Cores for Volatile and Semi-volatile Analysis	4	1/01	FINAL
G3-VOA-6	Procedure to Preserve Samples for Volatile Organic Analysis of Solid Matrices by Method 5035	2	1/01	FINAL
G3-VOA-7	Volatile Organic Preparation of Solid Matrices by Methods 5035	2	1/01	FINAL
G3-VOA-8	Volatile Compound Confirmation Under Wisconsin Administrative Code NR 700	4	1/01	FINAL
G3-VOA-9				RESERVED
G3-VOA-10				RESERVED
G3-VOA-11				RESERVED
G3-VOA-12				RESERVED
G3-VOA-13				RESERVED
G3-VOA-14				RESERVED
G3-VOA-15				RESERVED
G3-VOA-16				RESERVED
G3-VOA-17				RESERVED
G3-VOA-18	Analysis of Dissolved Methane, Ethane, Propane and Ethene in Ground Water by Static Headspace and Gas Chromatography	2	02/00	FINAL
G3-VOA-19				RESERVED
G3-VOA-20	Volatile Organic Preparation of Solid Matrices by Methods 5035/8260B - Perkin Elmer MS			ARCHIVED
G3-VOA-21	Tedlar Bag Preservation by Methanol	1	08/99	FINAL

SOP #	Title	Control		
	Volatile Organics (GRO)	Rev. #	Date	Status

G3-GRO-1	Soil/Semisolid Sample Preparation for the Analysis of Gasoline Range Organics and Petroleum Volatile Organics By Wisconsin Modified GRO	2	03/00	FINAL
G3-GRO-2	Modified Method For Determination Of Gasoline Range Organics	4	02/00	FINAL
G3-GRO-3	Method for determination of volatile petroleum hydorcarbons-gasoline-Iowa	3	05/00	FINAL
G3-GRO-4	Sample Sreening volatile organics prior to preparation	2	03/00	FINAL
G3-GRO-5	Total petroleum hydrocarbons-gasoline	5	1/01	FINAL
G3-GRO-6	Method 8021B Aromatic and halogenated volatiles by gas chromatography using photoionization detectors	5	1/01	FINAL
G3-GRO-7	Aqueous Sample preparation for the Analysis of Gas Range organics and petroleum volatile organics	2	02/00	FINAL

SOP #	Title	Control		
	SEMIVOLATILES (SVO)	Rev. #	Date	Status
G3SVO-1	WI Modified DRO	4	02/00	Final
G3SVO-2	Analysis of Polynuclear Hydrocarbons by HPLC	5	02/01	Final
G3SVO-3	Total Petroleum Hydrocarbons Modified GC	5	02/01	Final
G3SVO-4	Analysis of Polynuclear Hydrocarbons in Soil by GC/MS - SIMs Mode	4	02/01	Final
G3SVO-5	Method for Determination of Extractable Petroleum Hydrocarbons - Diesel Iowa	4	02/00	Final
G3SVO-6	Cleaning Glassware Used in the Analysis of Semivolatile Range Organics	4	02/00	Final
G3SVO-7	Determination of DRO Sample Mass and Methylene Chloride Addition	4	02/00	Final
G3SVO-8	Aqueous Sample Preparation for the Analysis of Polynuclear Aromatic Hydrocarbons HPLC and GC/MS	5	02/01	Final
G3SVO-9	Solid/Semisolid Sample Preparation for the Analysis of Polynuclear Aromatic Hydrocarbons HPLC and GC/MS	4	02/01	Final
G3SVO-10	TNRCC C_6 to C_{28} Petroleum Hydrocarbons	2	11/98	Final
G3SVO-11	Petroleum Range Organics - Florida	0	12/98	Final
G3SVO-12	Extractable Petroleum Hydrocarbons - Tennessee	0	01/99	Final
G3SVO-13	Analysis of Polynuclear Aromatic Hyrdrocarbons in Aqueous Matrices by GC/MS	3	02/01	Final
G3SVO-14	Analysis of Organophosphorus Pesticides	0	09/99	Archived
G3SVO-15	Aqueous Sample Preparation for the Analysis of Arochlor PCBs by GC	2	02/01	Final
G3SVO-16	Solid/Semisolid Sample Preparation for the Analysis of Arochlor PCBs by GC	2	02/01	Final
G3SVO-17	Sulfuric Acid Clean Up for the Analysis of Arochlor PCBs by GC	2	02/01	Final
G3SVO-18	Analysis of Polychlorinated Biphenyls by GC	1	02/01	Final

G3SVO-19	Analysis of Base Neutral and Acid Compounds by GC/MS - Full Scan	2	02/01	Final
G3SVO-20	Aqueous Sample Preparation for the Analysis of Base Neutral and Acid Compounds by GC/MS - Full Scan	1	02/01	Final
G3SVO-21	Solid/Semisolid Sample Preparation for the Analysis of Base Neutral and Acid Compounds by GC/MS - Full Scan	1	02/01	Final

SOP #	Title	Control		
	METALS (MET)	Rev. #	Date	Status
G2-MET-00	Acid Digestion of Waters for Dissolved or Total Recoverable Metals-ICP	1	02/01	Final
G2-MET-01	Acid Digestion of Aqueous samples and extracts for Total Metals - ICP and ICP-MS	1		Archived
G2-MET-02	Acid Digestion of Aqueous Samples for Total Metals Analysis by ICPMS	4	02/01	Final
G2-MET-03	Sample Preparation Procedure For Determination Of Total Recoverable Elements	3		Archived
G2-MET-04	Sample Preparation Procedure For Determination Of Arsenic and Selenium	3		Archived
G2-MET-05	Cleaning Metals Glassware	4	02/01	Final
G2-MET-06	Acid Digestion of Aqueous samples, Mobility-procedure extracts, and Wastes that contain suspended solids	4	02/01	Final
G2-MET-07	Acid Digestion of Sludges,Sediments,Soils and Oils for Total Metals	4	02/01	Final
G2-MET-08	The Determination of Trace Metals by a Perkin Elmer Zeeman 5100 GFAA	3		Archived
G2-MET-09	The Determination of Trace Metals by a Simultaneous TJA 61e Inductive Argon Plasma	3		Archived
G2-MET-10	Acid Digestion of Sludges,Sediments and Solid Waste Soils andCompositional Samples	3		Archived
G2-MET-11	Turbidity (Nephelometric)	1	02/01	Final
G2-MET-12	Acid Digestion of Solid Samples - Non Microwave Digestion	1	02/01	Final
G2-MET-13	Acid Digestion of Solid Samples for Silver or Antimony (Non-Microwave)	1	02/01	Final
G2-MET-14	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy	1	02/01	Final
G2-MET-15	Spiking Procedure for Metals Analysis	1	02/01	Final
G2-MET-16	Hardness Calculation	4	02/01	Final

SOP #	Title	Control		
	WET CHEMISTRY (WCM)	Rev. #	DATE	Status
G1-WCM-1	Pensky-Martin Closed-Cup Method For Determining Ignitability	2	10/98	FINAL
G1-WCM-2	Residue, Non-Filterable (Gravimetric, Dried)	2	10/98	FINAL
G1-WCM-3	pH Measurement in Environmental Samples	3	12/98	FINAL
G1-WCM-4	Total Solids Determination	2	12/98	FINAL

SOP #	Title	Control		
	CUSTOMER SERVICE (CS)	Rev. #	Date	Status

G1-CST-01	Conflict Resolution	2	10/98	Final
G1-CST-02	Desemmination Of Project Specific Requests	0	10/98	Final

SOP #	Title	Control		
	SAMPLE RECEIVING (REC)	Rev. #	Date	Status
G1-REC-01	Balance Calibration and Tare Weight of 60 mL (2oz.) containers	2	10/98	Final
G1-REC-02	Sample Bottle Tags	0	10/98	Final
G1-REC-03	Sample Bottle Request and Preparation	0	10/98	Final
G1-REC-04	Subcontracting	0	10/98	Final
G1-REC-05	Sample Receipt and Non Conformance Documentation Phone Logs	0	10/98	Final
G1-REC-06	Sample Labels Generated From The Labels Program	0	10/98	Final
G1-REC-07	Sample Receipt and Log-in	0	10/98	Final
G1-REC-08	Chain-of-Custody	2	10/98	Final
G1-REC-09	Maintenance of Ice Chests and Shipping Containers	0	01/99	Final

SOP #	Title	Control		
	QUALITY ASSURANCE (QAU)	Rev. #	Date	Status
G1-QAU-01	Initial Validation		02/00	Final
G1-QAU-02	Statistical Analysis of Method Detection Limit Studies (MDL Studies)		12/98	Final
G1-QAU-03	Determining and Reporting Significant Figures		12/98	Final
G1-QAU-04	Calibration of Balnaces and Weight Sets		05/00	Final
G1-QAU-05	Quality Assurance Facility Audit Schedule and Procedure		02/01	Final

SOP #	Title	Control		
	GENERAL (GEN)	Rev. #	Date	Status
G1-GEN-01	Reserved			
G1-GEN-02	Reserved			
G1-GEN-03	Reserved			
G1-GEN-04	Data Recording, Signatures, Dates, and Data Handling	0	09/99	Final
G1-GEN-05	Sample Disposal	1	12/98	Final
G1-GEN-06	Reserved			
G1-GEN-07	Reserved			
G1-GEN-08	Reserved			
G1-GEN-09	Purchasing/Supply Request/Receiving of Supplies	1	06/99	Final
G1-GEN-10	Reserved			
G1-GEN-11	Reserved			
G1-GEN-12	Hazardous Waste Handling Procedures and Emergency Response	1	06/99	Final
G1-GEN-13	Reserved			
G1-GEN-14	Sample Acceptance Policy	0	02/01	Final
G1-GEN-15	Nonconformance Memos	0	02/01	Final
G1-GEN-16	Reserved			
G1-GEN-17	Reserved			
G1-GEN-18	Reserved			
G1-GEN-19	Reserved			
G1-GEN-20	Reserved			
G1-GEN-21	Reserved			
G1-GEN-22	Reserved			
G1-GEN-23	Policy for Confidentiality and Release of Project Information	0	02/01	Final
G1-GEN-24	Reserved			
G1-GEN-25	Reserved			
G1-GEN-26	Reserved			
G1-GEN-27	Reserved			
G1-GEN-28	Reserved			
G1-GEN-29	Resolution of Client Complaints	0	02/01	Final

SOP #	Title	Control		
	DOCUMENT (DOC)	Rev. #	Date	Status
G1-DOC-01	Definitions of Organizational Responsibilities at En Chem Green Bay	0	10/98	Final
G1-DOC-02	Policy For The Release Of Data	0	10/98	Final
G1-DOC-03	Records Retention	2	10/98	Final
G1-DOC-04	Logbook Assignment	0	02/00	Final

SOP #	Title	Control		
	LABORATORY (LAB)	Rev. #	Date	Status
G1-LAB-01	Preparation of Standard Operating Procedures	1	10/00	Final
G1-LAB-01A	Pipettor Calibration	0	09/99	Final
G1-LAB-02	Minimum Quality Control Requirements For Sample Preparation And Analysis	0	10/98	Final
G1-LAB-03	Production and Monitoring of Reagent Grade Water	2	10/98	Final
G1-LAB-04	Standards Documentation	2	10/98	Final
G1-LAB-05	Label Removal	0	11/98	Final
G1-LAB-06	Charting of Recoveries in Control Samples	0	09/99	Final
G1-LAB-07	Reserved			
G1-LAB-08	Acknowledgment of Exposure to and Revision of Standard Operating Procedures (SOPs)	2	05/00	Final
G1-LAB-09	Traceability of Laboratory Reagents	0	12/99	Final
G1-LAB-10	Thermometer/Temperature Calibration	2	07/97	Final
G1-LAB-11	Reserved			
G1-LAB-12	Refrigerator and Freezer - Temperature Monitoring	0	01/01	Final
G1-LAB-13	Drying Ovens - Temperature Monitoring	0	01/01	Final

SOP #	Title	Control		
	INFORMATION SERVICE (LIS)	Rev. #	Date	Status

G1-LIS-01	Novell Backups	0	06/00	Final
G1-LIS-02	NT Backups	0	11/99	Final
G1-LIS-03	Data Archival of Hpchem, Hplcchem, ICPMS, and Target Data	0	11/99	Final
G1-LIS-04	Reserved			
G1-LIS-05	Reserved			
G1-LIS-06	Reserved			
G1-LIS-07	Reserved			
G1-LIS-08	Reserved			
G1-LIS-09	Reserved			
G1-LIS-10	Policy for Requested Changes to Electronic Records	0	10/99	Final

APPENDIX K

METHOD REFERENCES

En Chem, Inc. Method References

1. Federal Register, Volume 44, No. 223, December 3, 1979, 40 CFR Part 136, pp. 69464 to 69575.

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.

7. "Evaluation of Selected Lipid Methods for Normalizing Pollutant Bioaccumulation", Environmental Toxicology and Chemistry, Volume 10, 1992

8. "Official Methods of Analysis of the Association of Official Analytical Chemists," Methods Manual, 17th ed.

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.

12. The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils," Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, USEPA, Cincinnati, Ohio, April 1981.

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).

15. USEPA Contract Laboratory Program Statements of Work (CLP-SOW) OLM01.8, OLM03.1, and ILM04.0

EN CHEM INC.

...chemistry for the environment.

En Chem, Inc.
Green Bay Laboratory
Statement of Qualifications

Corporate Office & Laboratory
1795 Industrial Drive
Green Bay, WI 54302
Phone: (920) 469-2436 • Fax: (920) 469-8827
Toll Free: 800-736-2436

Madison Office & Laboratory
525 Science Drive
Madison, WI 53711
Phone: (608) 232-3300 • Fax: (608) 233-0502
Toll Free: 888-536-2436

www.enchem.com

STATEMENT OF QUALIFICATIONS

Revision 6.0 - December 2000

En Chem, Inc.
1795 Industrial Drive
Green Bay, WI. 54302
800-736-2436
EPA # WI00912

EN CHEM GREEN BAY FACILITY QUALIFICATIONS
REV. 5.0 AUGUST 2000

Prepared by: M. Guha

Title: Laboratory QA Coordinator

Approved: Nb K Melbey
Title: Operations Manager

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1.0 Introduction

1.1History

En Chem, Inc. is an environmental laboratory which was established in February of 1992. It was designed from the beginning to be highly automated, staffed with experienced analysts, and focused on a new level of customer service. Our staff has grown to 42 full time and 8 part time employees - as of February 1, 1997 in Green Bay.

En Chem, Inc. has established laboratory operation in September 1995 at En Chem Superior, Wisconsin.

En Chem, Inc. has established laboratory operation in April 1996 at En Chem Madison, Wisconsin at two locations. In the first quarter of 1998 the two locations were merged into one facility.

1.2 OVERVIEW OF EN CHEM AND STATEMENT OF POLICY

Our goal at En Chem Inc. is to provide our clients with quality services and total client support in environmental analysis and field sampling. All employees are committed to the quality assurance (QA) program described in the QA Plan. This not only includes scientific accuracy of results, but also rapid delivery of results.

With this goal in mind, the En Chem facility has been organized to process each sample for analysis with the highest amount of efficiency possible. En Chem has developed an electronic chain of custody support, field sampling kits to take soil samples and data management support services that will assist the client in collecting a proper sample. Most of the laboratory procedures have been electronically linked to LIMS to reduce errors and increase efficiency.

En Chem has developed Mobile laboratory capabilities to perform Diesel Range Organics (DRO), Gasoline Range Organics (GRO), PVOC and individual Volatile Organic Compounds (VOC). In order to provide quality analysis, En Chem has provided the mobile laboratory with the same state of the art equipment which is seen in a fix laboratory operation. En Chem developed these capabilities to provide the client with quality analysis consistent with normal fixed laboratory operation. In 1997 the mobile lab is developed capabilities to perform Pesticide,Nitrate-Nitrite,Nitrate and Ammonia analysis.

2.0 Facility

2.1 Laboratory Equipment

2.1.1 SEMI-VOLATILE AREA

4-Hewlett-Packard 5890 II GC with dual FID detectors
1-Hewlett-Packard 5890 II GC with FID detector
1- Hewlett-Packard 1090 LC with 1046A Fluorescence detector
1- Hewlett-Packard 5890 GC with 5972 MSD
1- Dionex ASE-200 Accelerated Solvent Extractor
2-Hewlett-Packard 6890 GC with 5973 MSD
1- Hewlett-Packard 5890 II GC with single ECD detector and TDX EZ Flash

All the Hewlett-Packard GC/MS systems have HP-ChemstationTM TargetTM software.

2.1.2 VOLATILE AREA

GC

7 - Hewlett-Packard 5890 II GC with PID/FID detectors
7- Hewlett-Packard 5890 II /Headspace GC with FID
4- Tekmar 3000 Purge and Trap Concentrators
2- Dynatech PTA-30 Autosamplers
3- Dynatech DynaTrap Purge and Trap and Autosampler System
2- Archon Autosampler

GC/MS

5 - Hewlett-Packard 5890 II GC with 5972 MSD
5 - Tekmar 3000 Purge and Trap Concentrators
2 - Archon Autosampler
3 - Dynatech PTA-30 Autosamplers

All the Hewlett-Packard GC/MS systems have HP-ChemstationTM TargetTM software.

2.1.3 METALS AREA

Graphite Furnance: Perkin Elmer 5100Z with Perkin Elmer 3030B
ICPMS-HP 4500

2.2 Floor Plan

2.2.1 CORPORATE HEADQUARTERS

En Chem, Inc. corporate headquarters is at 1241 Bellevue Street, Green Bay, WI, 54302.

2.2.2 ANALYTICAL FACILITIES

En Chem's main laboratory in Green Bay, Wisconsin, is divided into two separate buildings. The volatiles and metals sections are located at 1795 Industrial Drive and the Semi-volatile is located at 1241 Bellevue Street. The volatile section is in a separate building to minimize contamination from common laboratory solvents. All of the metals digestion and instrumentation is located in same adjacent area. En Chem, Inc. has 15000 sqft of laboratory space.

2.2.3 ASSOCIATED FACILITIES

En Chem operates another main analytical facility:

En Chem - Madison
525 Science Drive
Madison, WI 53711

En Chem has technical sales offices in the following locations:

En Chem - Superior
1423 North 8th Street
Superior, WI 54880

Northern Illinois Area
410 W. Main Street
Cary, Il. 60013

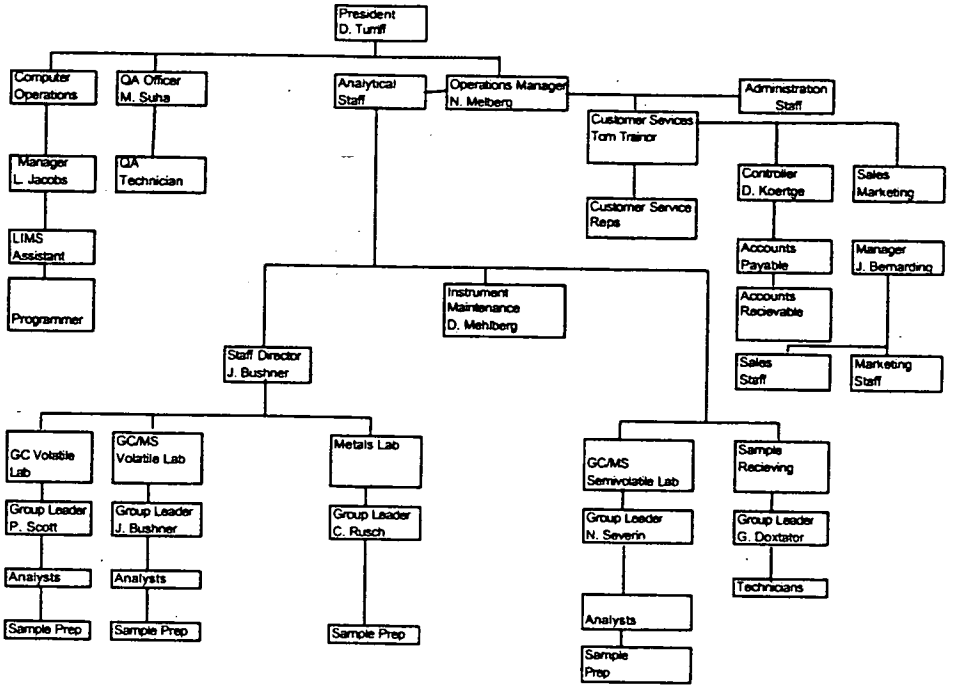
Central Wisconsin Area
315 Main Street
Mosinee, WI 54455

Greater Minneapolis Area
11900 Wayzata Blvd.
Suite 116F
Minnetonka, MN 5530.

En Chem - Ohio
4083 Sunset Drive
Medina, OH 44256

3.0 Personnel

3.1 Organizational Chart



LABORATORY PERSONNEL

3.2 Administrative Staff

Name	Degree, Field	Yrs. Exper.	Yrs, Env Lab
Nils K. Melberg	BS-Water Chemistry	14	14
Michael C. Suha	BS-Zoology	12	11
David E. Turriff	Ph.D-Biochemistry	23	15
Jill A. Duranceau	BS-Forestry	16	12
Ron Sommerhalder	BS-Mathematics	37	5

3.3 Volatiles

Name	Degree, Field	Yrs. Exper.	Yrs, Env Lab
Jeff Bushner	BS-Geology	13	12
Randy Naidl	BS-Chemistry	8	8
Phil Scott	BS-Water Resources/Chemistry	14	14
Hong Wiley	BS-Chemistry	11	9
Dennis Mehlberg	BS-Chemistry	26	10
Timothy L. Theisen	BS-Geology	16	13
Scott Turner	BS - Water Chemisrty	5	5

3.4 Semivolatiles

Name	Degree, Field	Yrs. Exper.	Yrs, Env Lab
Nick Severin	BS-Chemistry	7	7
Doug Basten	BS-Biology	7	7
Alan Orr	BS-Biology	7	7
Andrew Schleis	BS - Chemistry	5	2

3.5 Metals

Name	Degree, Field	Yrs. Exper.	Yrs, Env Lab
Chad Rusch	BS-Chemistry	4	4

3.6 Sample extraction and preparation

Name	Degree, Field	Yrs. Exper.	Yrs, Env Lab
Donavon Seiloff	BS- Chemistry	11	2
Dennis Monfort	BS - Biology	2	2
Sonja Stenli	BS-Environmental Sciences	4	2
Valarie Renquin	HS	22	4
Kate Grams	BS-Soil Sciences	5	2
Janis Ikaunieks	MS- Chemical Engineering	9	3

3.7 Sample Receiving

Name	Degree, Field	Yrs. Exper.	Yrs, Env Lab
Timothy A. Novoselatz	BS-Environmental Sciences	6	6
Gloria Doxtator	None	25	12
Phil Giovingo	None	45	4
Laura Ikauniece	MS -Environmental Engineering	8	3

3.8 Client Services

Name	Degree, Field	Yrs. Exper.	Yrs, Env Lab
Tom Trainor	BS-Chemical Engineering	18	13
Laurie Woelfel	None	23	13
Eric Bullock	BS-Environmental Sciences	3	3

3.9 Purchasing

Judy Theys	BS-Chemistry	17	12

3.10 Computer Services

Name	Degree, Field	Yrs. Exper.	Yrs, Env Lab
Lloyd Jacobs	None	27	13
John Kirsch	AS- Microcomputers	3	3
Kendall Hull	MS - Paper Science & Eng.	5	1

3.11 Resumes

David Turriff, Ph.D.

En Chem, Inc.
1241 Bellevue St.
Green Bay, WI 54302

1992-Present President, En Chem, Inc. Started the laboratory in 1992. Under his management, the laboratory has grown to over eight million in annual revenue with a staff of over 100. The laboratory has been featured in several national publications as a model of efficiency and management due largely to the automation of data handling and the low (<2% staff turnover). Three patents have been issued to En Chem relating to soil sampling methods. SW846 research results from studies undertaken at En Chem have been presented to seven state and federal agencies and have resulted in thirteen national presentations in the last two years.

1990-1992 Director of Toxicology, Bellin Memorial Hospital. Assisted the hospital in achieving certification by the National Institute on Drug Abuse. Bellin Laboratories achieved certification on its first inspection and was one of only sixty laboratories to be certified at the time.

1987-1990 Director, Foth and Van Dyke and ORTEK. Brought laboratory from $150,000 to $1 million in three years. The laboratory was sold to the Oneida Tribe of Indians in 1989. I managed this laboratory for two years, obtaining certification from the US Army Corp and Navy on the first attempt.

1984-1987 Supervisor of Organics, RMT Laboratory. Started the organics laboratory for RMT and, in the second year of operation, managed a major PCB project for General Motors that involved the analysis of over 1200 PCB samples.

1978-1984 Associate Director of Toxicology and Assistant Professor of Neurochemistry at the University of Illinois, Medical Center at Rockford. Involved in teaching of second year medical students and in research into mechanism of the chemistry of brain cell differentiation. Also involved in managing the environmental chemistry division of the toxicology lab.

1974-1978 Research Associate, University of Chicago, Department of Neurosurgery. Involved in basic research in the field of glial cell maturation.

EDUCATION

1976 Ph.D. Biochemistry/Neurochemistry University of Rochester, Rochester, New York.
1969 B.S. Chemistry, St. Norbert College, De Pere, Wisconsin

Nils K. Melberg

En Chem, Inc.
1241 Bellevue St.
Green Bay, WI 54302

1996-Present Operation manager, En Chem, Inc. Coordinate analysis of environment samples. Project equipment requirements and supplies needed to analyze a large volume of samples. Keep laboratory staff focused on a quality control program that will meet legally defensible data and quality objectives. Technical review of results and report signing. Review of all personnel responsibilities.

1993-1996 Quality Assurance Officer, En Chem, INC. Implemented a quality control program for analysis of DRO, GRO, Metals, Volatile and Semivoliltes. A 1994 WDNR audit of the laboratory resulted in no deficiencies and no non-compliances. The laboratory is certified in Wisconsin, Minnesota and is pending certification in Iowa and North Carolina.

1988-1993 Senior Chemist, En Chem, INC. Involved in the start up in 1992. Coordinated analysis of environmental samples. Analyzed samples utilizing GRO, DRO and VOC methodology. Method development of GRO and Volatile analysis.

1988-1992 Metals supervisor, Ortek, INC. Coordinated sample digestion and scheduled analysis utilizing the techniques of atomic absorption and inductively coupled argon plasma spectroscopy. Implemented methods for following CLP protocol. Accomplished method development of the ICP. Obtained certification from US Army Corp, Navy and CLP.

1985-1988 Chemist, Foth and Van Dyke. Responsible for metals analysis using flame, graphite furnace, hydride and cold vapor atomic absorption techniques. Developed graphite furnace analysis, and developed several auto analyzer methods. Other responsibilities included completing a variety of wet chemistry test.

EDUCATION

1984 B.S. Water Chemistry, UWSP, Stevens Point, Wisconsin

Jeffrey J. Bushner

En Chem, Inc.
1241 Bellevue St.
Green Bay, WI 54302

1994-Present GC/MS Group Leader, En Chem, Inc. Duties include operating and maintaining HP 5972 GC/MS for volatiles analysis as well as scheduling daily operation of GC/MS volatiles department. Troubleshooting, method's development and CLP hardcopy deliverables are also my responsibility.

1991-1994 Organic Manager, Precision Analytical Laboratory, Inc. Duties include daily operation of organic chemistry department; operating and maintaining HP 5970 and HP 5972 GC/MS for volatile and semivolatile methods according to EPA and CLP protocols. Producing CLP data deliverables for VOA, SVOA, and PCB/Pest; operating and maintaining Waters HPLC for PAH's (8310) and explosives (8330). Also was the technical interface between the laboratory and clients.

1989-1991 Organic Supervisor, ORTEK. Duties include daily operation of organic chemistry department; operating and maintaining Finnigan Mat Incos 50 GC/MS for semivolatiles according to EPA and CLP protocols; data reduction and collation of CLP data deliverables; technical data review before final laboratory reporting.

1987-1989 Organic Chemist, Interpoll Laboratories, Inc. Duties include operation and maintenance of Finnigan Mat Incos 50 GC/MS for Semivolatiles according to EPA and CLP protocols. GC analysis of PCB/Pest, including certification of PCB's by solid phase extraction by USATHAMA, air analysis by NIOSH and PCAM methods for volatiles.

1986-1987 Environmental Chemist, Texas Instruments, Inc. Duties include operation and maintenance of ARL 3520 ICAP for RECRA metals, PE5100 GFAA for As, Se and cold vapor Hg, Dionex 2000 for anions/cations; HP5970 GC/MS for semivolatiles and Finnigan Mat 4500 GC/MS for volatile methods by EPA SW846. Wet chemistry for cyanide, BOD, COD, fluoride, chloride, etc....

1985-1986 Environmental Chemist, Production Profits, Inc. Duties include operation of fuels testing lab for physical properties of crude oil and aviation fuels; wet chemistry on water and waste water according to Standard Methods; GC analysis of chlorinated hydrocarbons on crude oil (F500) on HP5880.

EDUCATION

1985 B.S. Geology, University of Wisconsin-Eau Claire EauClaire,WI

Phil Scott

En Chem, Inc.
1241 Bellevue St.
Green Bay, WI 54302

1993-Present GC Team Leader, En Chem Inc. Green Bay, WI.
Responsible for the coordination, extraction and daily work flow of 5 GC instruments analyzing WDNR Modified GRO/PVOC and other GC methods . Work directly with four staff members promoting an overall team concept. Assist computer personel in developing electronic data transfer and backlogs. Other duties include trouble-shooting and maintenance on all analytical instrumentation, method developement, and working with management staff.

1986-1993 Analyst/Supervisor, Foth and Van Dyke and ORTEK, Green Bay, WI. Analyzed a large variety of wet chemistry tests including cyanide, phenols, BOD, COD, BTU's, SO4, PO4, and all the various types of solid tests. Also did metals by FLAA, GFAA, and Hg by cold vapor techniques. The Foth and Van Dyke laboratory was sold to the Oneida Tribe of Indians in 1989 and was called Ortek. At Ortek, I analyzed metals using ICP and the above mentioned techniques. I supervised the metals staff of 5 people for almost 1.5 years. I was responsible for the daily work flow and coordination of two ICP's, 3 AAGF units and one Hg unit. We ran protocols and became certified for CLP, USCOE, DOE, DOD, NAVYHAZRAP, and general Army and Navy certifications. Other duties included trouble-shooting and maintenance, method developement, and working with management staff.

1985-1986 Analyst, CBC AquaSearch, Oak Creek, WI.
Performed a large variety of wet chemistry testing most of which are mentiond above.
Started some metals testing.

1985 Oshkosh Wastewater Treatment Facility, Oshkosh, WI.
Duties included lab testing and sampling of influent, effluent and sludge samples from various stages in the treatment process. Test results generated were used to run the facility and for reporting to the WDNR.
Hired as a temporary employee.

EDUCATION

1984 B.S. Water Resources - Chemistry Option. University of Wisconsin - Stevens Point

Chad Rusch

En Chem, Inc.
1241 Bellevue St.
Green Bay, WI 54302

1999-Present Metals Analysis Leader, En Chem Inc. Green Bay, WI.
Duties include operating and maintaining HP ICPMS for metals analysis as well as scheduling daily operation of metals department. Troubleshooting, method's development and CLP hardcopy deliverables are also my responsibility.

1999-1999 Technical Service Representative , Plastic Protective Coatings, Inc. Green Bay, WI.
Duties included troubleshooting customer problems in the applications of Plastite coatings over the phone and on -site. Mediated between contractors using coating products and end-client. Ran lab tests to verify product compatability with application.

1995-1999 Analyst Sample Preparation and Digestion, En Chem Inc. Green Bay, WI.
Experienced in separatory funnel, sonication, and accelerated solvent extractions for the analysis of semivolatile section. Method Development extraction and analytical procedures for Orgâno-Phosphorus compounds by GC/NPD.GFAA analysis using Perkin Elmer 5100 with Zeeman correction along with digestion metal samples using various SW846 and EPA procedures.

EDUCATION

199 B.S. Chemistry University of Wisconsin - Green Bay

Michael C. Suha

En Chem, Inc.
1241 Bellevue St.
Green Bay, WI 54302

1995-Present Quality control technician, En Chem, Inc. Assisted in the review of final reports to clientel. Promotion to Quality Assurance Coordinator responsible for performance evaluations by laboratory section and laboratory certification. New certifications acheived for the Green Bay facility were: Iowa ,North Dakota and South Carolina. Presently on the Board of Directors of the Wisconsin Environmental Laboratory Association. Also serves on the Technical Adivisory Committee which reports to the Wisconsin Department of Natural Resources on issues affecting laboratory accreditation issues.

1991-1995 Project Manager, Robert E. Lee & Associates, Inc.Overseeing and coordinating analytical services for clients utilizing EPA and Wisconsin Natural Resource Codes. Researching specialized analytical applications for Non-regulatory projects.

1990-1991 National Client Services Manager,Ortek Environmental Laboratories . Proposal preparation, Lab scheduling, Project tracking, Final report generation and transmission

1990-1991 Client Services Representative,Heritage Laboratories, Inc. Quotation preparation, Sampling crew scheduling,sample receiving, sample documentation and maintenance.

1989-1990 Field Chemist,Laidlaw Environmental Services. Establishing and implementing house keeping rules for a Transfer Station at a government facility with co-workers under the direction of EPA and military officials. Lab packing, Field characterization of waste streams,equipment maintenance and trouble shooting

1988-1989 Metals analyst,Johnson Controls, Inc. Effluent wastewater analysis for Globe Union Battery production facilities nationwide.

EDUCATION

1988 BS Zoology 1988 University of Wisconsin - Milwaukee

Nickolas J. Severin

En Chem, Inc.
1241 Bellevue St.
Green Bay, WI 54302

1992-Present Semivolatile Section Leader, En Chem, Inc. Responsible for sample thoroughput and quality control in the semivolatile section. Analyzed soil and water samples for DRO, GRO, PVOC, PCB, Pesticide, PAH and semivolatile analytes using GC, HPLC, and GC/MS instrumentation. Knowledgeable in ECD, FID, PID, UV-DAD, Fluorescence, and Mass Spectrometer detection systems. Experienced in separatory funnel, sonication, and accelerated solvent extractions. Supervised analysts and extraction personnel and monitored supply ordering for the the semivolatile section. Developed extraction and analytical procedures for the semivolatile section.

1991-1992 Laboratory Assitant, Environmental Task Force Laboratory. Extracted soil and water samples for pesticides, trace organics and herbicides. Performed PCB analyses on fish from the Lake Michigan region. Used soxhlet extractions and a variety of cleanup procedures in these analyses.

1991 Research Assistant, University of Michigan - Ann Arbor. Synthesized and studied organometallic compounds in Dr. Coucouvanis' Research Group. Experimented under nitrogen atmospheres with glove boxes.

1989-1991 Stockroom Assistant, University of Wisconsin - Stevens Point Chemistry Stockroom. Handled the stocking and distribution of chemicals and chemical supplies. Checked laboratory safety devices for defects. Prepared standard stock solutions.

EDUCATION

1992 BS-Chemistry, University of Wisconsin - Stevens Point

Lloyd Jacobs

En Chem, Inc.
1241 Bellevue St.
Green Bay, WI 54302

1992-Present - Manager of Information Systems, En Chem, Inc.
Responsible for the design, installation and maintenance of computer systems in the laboratory. Developed a system of automated data transfer from analytical instruments to the laboratory information management system, resulting in a significant boost in productivity and reduction in data transcription errors. In 1996 the Madison laboratory was upgraded to a windows based laboratory information system.

1989-1992 Computer Specialist, Ortek, Inc.
Installed, maintained, and customized a Novell network system allowing for the central storage of all lab data. Installed and customized a Laboratory Information Management System.

1986-1989 Systems and Database Administrator, Zimpro/Passavant, Inc.
Designed and programmed a Laboratory Information Management System for Enviroscan, Inc.

Authored and presented the paper "Automation of Laboratory Data Entry and Reporting Systems" at HAZMAT southwest September 1993.

Published "Automation of laboratory data entry systems" in the May 1994 issue of American Environmental Laboratory magazine.

Authored and presented the paper "Laboratory Data Management" at PITTCON '95 March 1995.

Thomas J. Trainor

En Chem, Inc.
1241 Bellevue St.
Green Bay, WI 54302

1995-present Project Management, En Chem, Inc. Responsibility is to represent client's interest on projects. Duties include: ensuring reasonable turn around time, contacting client with updates or problems, advising and troubleshooting for clients, expediting projects if requested, transmitting information as requested. Involved in project specific bid tabulations for clients.

1991-1995 Semivolatile Section Leader, Robert E. Lee & Associates Responsible for sample thoroughput and quality control in the semivolatile section. Analyzed soil and water samples for DRO, GRO, PVOC, PCB,Pesticide, PAH and semivolatile analytes using GC, HPLC, and GC/MS instrumentation. Knowledgeable in ECD, FID, PID, UV-DAD, Fluorescence, and Mass Spectrometer detection systems. Experienced in separatory funnel, liquid-liquid, sonication, and accelerated solvent extractions. Supervised analysts and extraction personnel and monitored supply ordering for the the semivolatile section. Developed extraction and analytical procedures for the semivolatile section.

1987-1991 Research Chemist, Hazelton Wisconsin, Inc. · Replaced laboratory manager during his absences. Managed off-site laboratory.Prepared budgets, cost estimates, cost analysis, capital expenditure requests and monthly financial reports. Interviewed, hired, trained, evaluated, fired and supervised personnel. Responsible for daily operations and production of 3 to 35 staff members both technically and financially. Performed final technical and administrative review of all reports and projects. Planned, lead and managed multiple complex projects in support of client registration of pesticides with the EPA. Acted as Study Director and Principal Analytical Investigator for as many as 10 projects at a time ranging in scope from 30 to 3,000 samples, $2,000 to $200,000 in value and 2 months to 24 months in duration. Coordinated group schedules to meet client, management, company and government requirements. Designed and wrote proposals, protocols, amendments, reports, methods and procedures for EPA and FDA registrations. Designed and implemented spreadsheets for raw data calculation using Lotus 123 and Excel. Designed and implemented standard report formats and standard narrative qualifications of analytical data. Provided clients with technical support and hosted client visits. Improved and standardized many complicated inter-departmental and intra-departmental processes. Responsible for monitoring analytical work to assure that the facilities, equipment, method, practices, procedures, records and controls are in conformance with state and federal regulations. Regulatory experience includes RCRA, FIFRA, TSCA, OSHA, CWA, CAA, CERCLA, CLP, GLP and LUST work. Comprehensive experience in HPLC utilizing UV, Fluorescence, Diode Array Detectors and GPC. Comprehensive experience in GC utilizing ECD, FID, NPD and TCD detectors. Performed extraction on many different matrices which included separation, clean-up and purification techniques. Performed method development, refinement and validation along with method and equipment trouble-shooting

EDUCATION

1986 BS Chemical Engineering, University of Wisconsin-Madison

4.0 Certifications

The Capabilities of the laboratory are as listed on the certification by parameter and program. Not all State Certification programs are equal. A summary page is also included for refernence. To be positive the laboratory can fullfill the analytical requirements of the project please call to discuss the particulars with a repersentative of En Chem.

Summary

STATE/AGENCY	AIR	UST	RCRA	CWA	SDWA	OTHER	CERTIFICATION#
Alabama		Accepted	Accepted	Accepted	X		
Alaska		X	X	X	X		
Arizona	X	X	X	X	X		
Arkansas		X	X	X	X		
California		X	X	X	X	X	
Colorado		Accepted	Accepted	Accepted	X		
Connecticut		X	X	X	X		
Delaware		Accepted	Accepted	Accepted	X	X	
Florida		X	X	X	X	**DEP Appr.[1,2]**	**CQapp 940300[1]** **CQapp 990073[2]**
Georgia		Accepted	Accepted	Accepted	X		
Hawaii		Accepted	Accepted	Accepted	X		
Idaho		Accepted	Accepted	Accepted	X		
Illinois		Accepted	Accepted	Accepted	X		
Indiana		Accepted	Accepted	Accepted	X		
Iowa	X	**CERT[2]**	Accepted	X	X		**135[2]**
Kansas		X	X	X	X		
Kentucky		Accepted	Accepted	Accepted	X		
Louisiana		**CERT[2]**	**CERT[2]**	**CERT[2]**	X		**AI85162[2]**
Maine		Accepted	Accepted	X	X		
Maryland		Accepted	Accepted	X	X		
Massachusetts		Accepted	Accepted	X	X		
Michigan		Accepted	Accepted	Accepted	X		
Minnesota		**CERT[1,2]**	**CERT[1,2]**	**CERT[1]**	**CERT[1]**		**055-999-107[1]** **055-999-334[2]**
Mississippi		Accepted	Accepted	Accepted	X		
Missouri		Accepted	Accepted	Accepted	X		
Montana		Accepted	Accepted	Accepted	X		

An 'X' indicates that the State/Agency has a program in place. **Laboratory NOT Certified.**
Accepted = The state does not have formal certification program.
CERT[1] = Madison Laboratory
CERT[2] = Green Bay Laboratory
Note! 'CERT' indicates Laboratory is Certified for Limited parameters. Check with Laboratory for specific

Summary - cont.

Nebraska		Accepted	Accepted	Accepted	X		
Nevada		Accepted	Accepted	X	X		
New Hampshire		Accepted	Accepted	X	X		
New Jersey		X	X	X	X		
New Mexico		Accepted	Accepted	Accepted	X		
New York		X	**CERT[1]**	**CERT[1]**	X	X	**11436**
North Carolina		**CERT[1]**	**CERT[1]**	**CERT[1]**	X		**503**
North Dakota		**CERT[1,2]**	**CERT[1,2]**	**CERT[1,2]**	**CERT[1,2]**		**R-159[1]** **R-150[2]**
Ohio		Accepted	Accepted	Accepted	X	X	
Oklahoma		**CERT[2]**	**CERT[2]**	**CERT[2]**	X		**9918[2]**
Oregon		Accepted	Accepted	Accepted	X		
Pennsylvania		Accepted	Accepted	Accepted	X		
Rhode Island	X	X	X	X	X		
South Carolina		**CERT[1,2]**	**CERT[1,2]**	**CERT[1]**	**CERT[1]**		**83001[1]** **83006[2]**
South Dakota		Accepted	Accepted	Accepted	X		
Tennessee		Approved[2]	Accepted	Accepted	X		
Texas		Accepted	Accepted	Accepted	X		
Utah		X	X	X	X		
Vermont		Accepted	Accepted	Accepted	X		
Virginia		Accepted	Accepted	Accepted	X		
Washington		X	Accepted	X	X		
West Virginia		X	X	X	X		
Wisconsin		**CERT[1,2]**	**CERT[1,2]**	**CERT[1,2]**	**CERT[1]**		**113172950[1]** **405132750[2]**
Wyoming		Accepted	Accepted	Accepted	X		
USACE		Accepted[2]				**Approved [1]**	

An 'X' indicates that the State/Agency has a program in place. **Laboratory NOT Certified.**
Accepted = The state does not have formal certification program.
CERT[1] = Madison Laboratory
CERT[2] = Green Bay Laboratory
Note! 'CERT' indicates Laboratory is Certified for Limited parameters. Check with Laboratory for specific

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. (Green Bay)
1795 Industrial Drive
Green Bay, WI 54302

405132750
Laboratory ID Number

Issued: August 29, 2000

Expires: August 31, 2001

for the following test categories:

- Physical
  - Total Solids
  - Total Suspended Solids
- General III
  - Waste Fingerprinting
  - Ignitability
- Metals I
  - Silver
  - Aluminum
  - Arsenic
  - Boron
  - Barium
  - Beryllium
  - Calcium
  - Cadmium
  - Cobalt
  - Chromium (Total)
  - Copper
  - Iron
  - Potassium
  - Magnesium
  - Manganese
  - Molybdenum
  - Sodium
  - Nickel
  - Lead
  - Antimony
  - Selenium
- Metals I
  - Strontium
  - Thallium
  - Vanadium
  - Zinc
- Metals II
  - Titanium
- Organics; Purgeable
  - Purgeable Aromatics
  - Purgeable Halocarbons
  - Volatile Organics (VOCs)
- Semivolatiles by GC/MS
  - Base/Neutral/Acid Extract
- Liquid Chromatography
  - PAHs by LC
- Petroleum Hydrocarbons
  - Diesel Range Organics
  - Gasoline Range Organics
  - Petroleum VOCs
- Organics; Organochlorine
  - PCBs

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

MINNESOTA MDH DEPARTMENT of HEALTH

Public Health Laboratory

Minnesota Department of Health

Certificate

In accordance with Minnesota Law and Rules
En-Chem, Inc.
1795 Industrial Drive, Green Bay, Wisconsin 54302
Laboratory Number: 055-999-334

has been certified for the analytes listed below and in our letter dated November 6, 2000.

Clean Water Program
Solids..Nonfilterable (TSS)
Metals
Volatile Organic Compounds
Synthetic Organic Compounds

Resource Conservation and Recovery Program
Metals
Volatile Organic Compounds
Synthetic Organic Compounds

Underground Storage Tank Program
Diesel Range Organics (DRO)
Gasoline Range Organics (GRO)
Petroleum Volatile Organic Compounds

Certification Expiration Date: August 31, 2001

Jan Malcolm, Commissioner of Health

MINNESOTA MDH DEPARTMENT OF HEALTH

Protecting, maintaining and improving the health of all Minnesotans

November 6, 2000

Michael C. Suha
En-Chem, Inc.
1795 Industrial Drive
Green Bay, Wisconsin 54302

RE: Laboratory Number: 055-999-334

Dear Mr. Suha:

We have received your laboratory's request for the addition of PCBs under the Resource Conservation and Recovery Program, 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 Wisconsin and Minnesota.

Laboratory Number: 055-999-334 Certification Expiration Date: 31-AUG-2001

Clean Water Program

Solids, Nonfilterable (TSS)	Vinyl chloride	Chlorobenzene
Aluminum	Bromomethane	1,1,1,2-Tetrachloroethane
Antimony	Chloroethane	Bromoform
Arsenic	Trichlorofluoromethane	1,1,2,2-Tetrachloroethane
Barium	1,1-Dichloroethene	1,2,3-Trichloropropane
Beryllium	Methylene chloride	Bromobenzene
Boron	trans-1,2-Dichloroethene	2-Chlorotoluene
Cadmium	1,1-Dichloroethane	4-Chlorotoluene
Calcium	2,2-Dichloropropane	1,3-Dichlorobenzene
Chromium	cis-1,2-Dichloroethene	1,4-Dichlorobenzene
Cobalt	Chloroform	1,2-Dichlorobenzene
Copper	Bromochloromethane	1,2-Dibromo-3-Chloropropane
Iron	1,1,1-Trichloroethane	1,2,4-Trichlorobenzene
Lead	Carbon tetrachloride	Hexachlorobutadiene
Magnesium	1,2-Dichloroethane	1,2,3-Trichlorobenzene
Manganese	Trichloroethene	Benzene
Molybdenum	1,2-Dichloropropane	Toluene
Nickel	Bromodichloromethane	Ethyl benzene
Selenium	Dibromomethane	m+p-Xylene
Silver	cis-1,3-Dichloropropene	o-Xylene
Sodium	trans-1,3-Dichloropropene	Styrene
Thallium	1,1,2-Trichloroethane	Isopropylbenzene
Vanadium	1,3-Dichloropropane	n-Propylbenzene
Zinc	Tetrachloroethene	1,3,5-Trimethylbenzene
Dichlorodifluoromethane	Chlorodibromomethane	tert-Butylbenzene
Chloromethane	1,2-Dibromoethane	1,2,4-Trimethylbenzene

Laboratory Number: 055-999-334
Certification Expiration Date: 31-AUG-2001

Clean Water Program Continued

sec-Butylbenzene
p-Isopropyltoluene
n-Butylbenzene
Naphthalene
Ethyl ether
Methyl ethyl ketone
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

Resource Conservation and Recovery Program

Arsenic
Barium
Cadmium
Chromium
Copper
Lead
Molybdenum
Nickel
Selenium
Silver
Zinc
Acetone
Carbon disulfide
Methyl ethyl ketone
Butyl benzyl phthalate
Di-2(ethylhexyl) phthalate
Di-n-butyl phthalate
Dimethyl phthalate
PCBs
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

Underground Storage Tank Program

Diesel Range Organics (DROs)
Gasoline Range Organics (GROs)
Petroleum Volatile Organic Compounds

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

STATE OF IOWA

TERRY E. BRANSTAD, GOVERNOR

DEPARTMENT OF NATURAL RESOURCES
LARRY J. WILSON, DIRECTOR

10/22/1998

En Chem, Inc.
1795 Industrial Drive
Green Bay, WI 54302

ATTENTION: Michael Suha, QA Officer
IA Lab #: 135
Expires: 07/01/2000

Dear Mr. Suha:

The Iowa Department of Natural Resources is pleased to grant CERTIFICATION for En Chem, Inc. in Green Bay, WI, to analyze environmental samples for reporting UST / WW results to the Iowa Department of Natural Resources.

This certificate supersedes any prior certificates issued from this Department.

You may not have received full certification for all of the parameters you requested or the parameter list may have changed since your last certificate was issued. Please review the parameter list and this certificate in detail to clarify the parameters for which your laboratory is certified.

The analytical methods that must be used are those shown on the attached parameter list and have been recommended for approval by the laboratory appraisal officer. Also attached is a listing of essential personnel and reporting requirements.

Certification by this Department is based upon the enclosed recommendation from the representatives of the University Hygienic Laboratory (UHL), current and future satisfactory performance on the audit samples for the listed parameters, and satisfactory performance throughout the certification period. If a change in certification status occurs during the certification period (i.e., due to a change in supervisory personnel, physical facility, methodology, performance on the audit samples, etc.), the laboratory must notify the Department in writing within 30 days of that change. Any questions may be directed to Stacy Freeburg, Laboratory Appraisal Coordinator, at 319/335-4500.

Use your assigned Lab ID (see top of page) when reporting results and when corresponding with this Department.

Be reminded that it is the laboratory's responsibility to keep this Department and UHL updated on any changes in current certification status in this laboratory's home state. It is also the laboratory's responsibility to automatically send to the Department and UHL the necessary and appropriate information required to keep this certification active and accurate without being prompted by either this department or UHL. Failure to do so may result in revocation of active certification in the State of Iowa.

A copy of this letter as well as the attachments should be made available to laboratory personnel for reference.

WALLACE STATE OFFICE BUILDING / DES MOINES, IOWA 50319 / 515-281-5145 / TDD 515-242-5967

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 - GREEN BAY
1795 INDUSTRIAL DR
GREEN BAY, WISCONSIN 54302

is hereby certified to perform analyses as documented on the attached parameter list(s). This certification does not guarantee validity of data generated, 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 and must be surrendered upon demand. This certificate is non-transferable and is valid only for the parameters and methodology listed on the attached parameter list(s).

Laboratory Director: NILS K MELBERG
Certifying Authority: WI
Date of Issue: November 22, 2000
Date of Expiration: August 31, 2001
Certificate Number: 83006001

R. Wayne Davis
Director
Office of Environmental Laboratory Certification

CR-000509 8/99

D H E C

PROMOTE PROTECT PROSPER

OFFICE OF ENVIRONMENTAL
LABORATORY CERTIFICATION
PO Box 72, State Park, SC 29147
(803) 896-0970 Fax (803) 896-0850

November 28, 2000

NILS K MELBERG
EN CHEM INC - GREEN BAY
1795 INDUSTRIAL DR
GREEN BAY, WISCONSIN 54302

Laboratory I. D. 83006

Dear Nils K Melberg:

Based upon your laboratory's remittance of all outstanding fees and/or updated certification information, I am pleased to enclose your amended certificate and associated parameter list. The parameter list should be compared with your laboratory's original application to determine if any parameters have been added or omitted and that the correct method reference is listed. If problems are detected, please contact this office within ten (10) working days.

Please be informed that these documents now represent the certificate of record for your laboratory. Any certificate(s) and associated parameter list(s) received prior to your receipt of these documents are now null and void and should be destroyed. Please be reminded that all environmental data submitted to the Department is reviewed to ensure that the reporting laboratory possesses the necessary certification. Data reported by laboratories without the proper certification will be addressed by the affected enforcement programs.

Any questions concerning the Laboratory Certification Program or the action(s) taken may be addressed to me. Please accept my congratulations regarding this achievement.

Sincerely,

R. Wayne Davis, Director
Office of Environmental Laboratory Certification
Bureau of Environmental Services

RWD:ic
Enclosures

SOUTH CAROLINA DEPARTMENT OF HEALTH AND ENVIRONMENTAL CONTROL
ENVIRONMENTAL LABORATORY CERTIFICATION PROGRAM

EN CHEM INC - GREEN BAY (Laboratory ID 83006)
Laboratory Director: NILS K MELBERG
Certifying Authority: WI
Certificate Number: 83006001

Date of Issue: November 22, 2000
Expiration Date: August 31, 2001

SOLID & HAZARDOUS WASTES

INORGANIC - TRACE METAL

ALUMINUM	EPA 6020
ANTIMONY	EPA 6020
ARSENIC	EPA 6020
ARSENIC	EPA 7060A
BARIUM	EPA 6020
BERYLLIUM	EPA 6020
CADMIUM	EPA 6020
CADMIUM	EPA 7131A
CALCIUM	EPA 6010B
CHROMIUM	EPA 6020
COBALT	EPA 6020
COPPER	EPA 6020
IRON	EPA 6010B
LEAD	EPA 6020
LEAD	EPA 7421
MAGNESIUM	EPA 6010B
MANGANESE	EPA 6020
MOLYBDENUM	EPA 6010B
NICKEL	EPA 6020
POTASSIUM	EPA 6010B
SELENIUM	EPA 6010B
SELENIUM	EPA 7740
SILVER	EPA 6020
SODIUM	EPA 6010B
THALLIUM	EPA 6020
THALLIUM	EPA 7841
VANADIUM	EPA 6010B
ZINC	EPA 6020

SEMI-VOLATILES

POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC	EPA 8310	EPA 3510C
POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC	EPA 8310	EPA 3545
POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC	EPA 8310	EPA 3580A
SEMIVOLATILE ORGANICS BY GC/MS:CAP. COL.	EPA 8270C	EPA 3510C
SEMIVOLATILE ORGANICS BY GC/MS:CAP. COL.	EPA 8270C	EPA 3545
SEMIVOLATILE ORGANICS BY GC/MS:CAP. COL.	EPA 8270C	EPA 3580A

VOLATILES (VOCS)

VOLATILE ORGANICS BY GC/MS: CAPILLARY COL.	EPA 8260B	EPA 5030B
VOLATILE ORGANICS BY GC/MS: CAPILLARY COL.	EPA 8260B	EPA 5035

STATE OF TENNESSEE
DEPARTMENT OF ENVIRONMENT AND CONSERVATION
Division of Underground Storage Tanks
4th Floor L&C Tower, 401 Church Street
Nashville, TN 37243-1541

August 4, 2000

Mr. Michael C. Suha
En Chem - Green Bay
1795 Industrial Drive
Green Bay, WI 54302-

Dear Mr. Suha:

Evaluation of all Division processes is currently underway. As a result, we will no longer maintain an "approved" laboratory list. Submittal of previously requested information to the Division is no longer necessary.

Please note that a change has occurred in the required analytical method for volatile organics. The current required analytical methods are;

Constituents	Method
Benzene, MTBE	8260A
TPH-GRO	TN Gasoline Range Organics Method
TPH-EPH	TN Extractable Petroleum Hydrocarbon Method

Copies of these methods can be obtained from the Division's website at www.state.tn.us/environment/ust/. Other program changes will be posted at this location as they occur. You are encouraged to routinely check this source for program updates.

The Division recently adopted a Quality Management Plan and is implementing it in all processes. You are encouraged to access EPA's website at www.epa.gov/quality/ to obtain updated information on national efforts. Of particular interest to you is the current efforts at laboratory accreditation. Information on this program can be obtained at www.epa.gov/ttn/nelac/. You are encouraged to participate in this program.

Sincerely,

Wayne Gregory

Wayne Gregory
Director
TN Division of Underground Storage Tanks

LWG:CAC:cmb

FLORIDA DEPARTMENT OF HEALTH

Jeb Bush
Governor

Robert G. Brooks, M.D.
Secretary

September 26, 2000

Michael C. Suha
En Chem, Inc.
1795 Industrial Drive
Green Bay, WI 54302

Subject: Quality Assurance Review; **En Chem, Inc.**; Comprehensive QA Plan # 990073, Revision # 1

Dear Mr. Suha:

The Statement of Intent and QA Planner file for the above referenced plan were received on August 25, 2000. The Department of Health (DOH), Laboratory Certification Program has completed the electronic evaluation of your plan and has approved it as explained in the attached evaluation report, with an effective date of September 26, 2000.

DEP will begin rulemaking to amend the QA Rule, Chapter 62-160, F.A.C. late this summer. Among the proposed changes will be the elimination of the CompQAP requirement and substitution of other requirements for laboratories and field operations. Because of these changes, all current CompQAP approvals are extended indefinitely until the elimination of the CompQAP requirement is promulgated by final rule.

Do not send an annual amendment or renewal letter for this CompQAP unless:

- The organization name has changed
- Amendments or revisions were specifically requested by DEP
- Amendments are required to work on a project regulated by DEP
- New sampling or laboratory capabilities have been added
- Alternative sampling or laboratory procedures are being proposed for approval

If amendments are submitted, they must be sent to the Department of Health's Laboratory Certification Program in Jacksonville. If you are submitting a plan using the QA Planner Software, the most current version of the FPC Software (dated 6/1/1998) must be used. This software may be downloaded from the internet at www.floridadep.org/labs/fpc.htm. Most current users already possess this version. To check the software version, launch the software, click on "Help", then choose "About QA Planner".

If you have any questions concerning this matter, please contact Mayra Quintana at (904) 791-1592 (voice) or Mayra_Quintana@doh.state.fl.us (email).

Sincerely,

Stephen A. Arms, Program Administrator
DOH, Laboratory Certification Program

For: Sylvia S. Labie, QA Officer
DEP, Quality Assurance Section

SAA/SSL/mq
Attachments: Evaluation Report
cc: Sylvia S. Labie, DEP, QA Section

State of Arkansas

Department of Environmental Quality
Laboratory Certification Program

Be it known that **En Chem, Inc.**
Green Bay, Wisconsin
has earned certification by this Department for the period of

August 3, 2000 to August 3, 2001

The following parameters are certified:

TPHC

August 3, 2000
Date

Jeffrey M. Pauls
Quality Assurance Officer

State of Louisiana

Department of Environmental Quality

M.J. "MIKE" FOSTER, JR.
GOVERNOR

J. DALE GIVENS
SECRETARY

CERTIFIED MAIL #Z509 781 684
Return Receipt Requested

June 2, 2000

AI #85162

Mr. Michael C. Suha
En Chem, Inc.
1241 Bellevue St., Suite 9
Green Bay, WI 54302

RE: Louisiana Application for Environmental Laboratory Accreditation

Dear Mr. Suha:

The Louisiana Department of Environmental Quality (Department) Laboratory Accreditation Program has completed a review of your application and at this time we do not need any further information. The Department will contact the State Accreditation Authorities in Wisconsin and elsewhere to determine whether the Accreditation you hold meets the requirements of the Department for the granting of reciprocal Accreditation. The Department will notify your Laboratory of the status of your application when such a determination has been made.

Please use the agency interest (AI) number on all future correspondence to the Louisiana Environmental Laboratory Accreditation Program (LELAP) and for use by the proficiency test provider. The AI number found directly below the date should be used as the Louisiana laboratory code number. Please place this number on the upper left-hand side of all correspondence below the date.

This number is not a certification number. Nor does the number imply certification. The AI number is used to designate your facility in the Louisiana Department of Environmental Quality database. The number is assigned to your laboratory by the Department's database. LELAP has no control in the assignment of the AI number.

At this time the Department is reviewing all applications. Laboratory audits will be scheduled after the initial application reviews are completed.

If you have any questions, please contact David Boucher at (225) 765-0582 or (225) 763-5515.

Sincerely,

Louis R. C. Johnson, Administrator
Laboratory Service Division

LRCJ: db

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. - 1795 Industrial Drive, Green Bay, Wisconsin
by
The State of Wisconsin Department of Natural Resources
for
All chemistry test categories and associated analytes for which they retain certification from the State of Wisconsin Department of Natural Resources

Certificate Number: R-150

Date of issue: November 1, 2000 Expiration Date: August 31, 2001 Covers: 11/1, 2000 - 8/31, 2001

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. Recognition of a laboratory's certification by the North Dakota Department of Health is not an endorsement or a guarantee of the validity of the results reported by the laboratory.

Myra Kosse
Director, Division of Chemistry

Earl Erickson
Certification Officer

MARK COLEMAN
Executive Director

OKLAHOMA DEPARTMENT OF ENVIRONMENTAL QUALITY

FRANK KEATING
Governor

December 6, 2000

Michael Suha
En Chem Green Bay
1241 Bellevue Street
Green Bay, WI 54302

Dear Mr. Suha:

We are pleased to inform you that the Oklahoma Department of Environmental Quality (ODEQ) has granted interim certification to your laboratory under the ODEQ's Laboratory Certification Program.

Enclosed is a list of parameters for which your laboratory is certified to perform analyses. This list fully demonstrates your laboratory's interim certification with the ODEQ for the period November 3, 2000 through August 31, 2001.

You must maintain on file this list of your laboratory's certified parameters. This means that at least one copy of the list should be kept available in your laboratory at all times for review by clients, the ODEQ, or approved ODEQ on-site inspectors.

If you have any questions concerning your certification please contact me at (405) 702-1024.

Sincerely,

Anthony R. Bright
Laboratory Certification Officer
Customer Services Division

707 NORTH ROBINSON, P.O. BOX 1677, OKLAHOMA CITY, OKLAHOMA 73101-1677

printed on recycled paper with soy ink

MARK COLEMAN
Executive Director

OKLAHOMA DEPARTMENT OF ENVIRONMENTAL QUALITY

FRANK KEATING
Governor

OKLAHOMA DEPARTMENT OF ENVIRONMENTAL QUALITY

INTERIM LABORATORY DUAL CERTIFICATION

FOR

GENERAL WATER QUALITY/SLUDGE TESTING

Issued in accordance with and pursuant to Title 27A, Section 2-4-301 of Oklahoma Statutes

En Chem _Green Bay 1241 Bellevue Street Green Bay, WI 54302 Michael Suha (920) 469-2436	Lab ID No.: 9918 Expires: 08/31/01 Issued: 11/03/00 Revised:

is hereby **CERTIFIED** for the parameters listed on the following page(s).

By acceptance of this certification, the laboratory agrees to operate and maintain its facilities in accordance with the state laboratory certification laws, 27A O.S. 1933 Supp. §§ 2-4-301 et. seq. and the rules and regulations adopted thereunder regarding the requirements for certified laboratories.

This certification is non-transferable and is valid only for the laboratory to which it is issued and valid only for the parameters listed on the following page(s).

Anthony R. Bright

Anthony R. Bright

Laboratory Certification Officer
Oklahoma Department of Environmental Quality

707 NORTH ROBINSON, P.O. BOX 1677, OKLAHOMA CITY, OKLAHOMA 73101-1677

printed on recycled paper with soy ink

OKLAHOMA
DEPARTMENT OF ENVIRONMENTAL QUALITY

State Environmental Laboratory
P.O. Box 1677
Oklahoma City, Oklahoma 73101-1677
405-702-6100

En Chem, Inc.- Green Bay Facility
ID # 9918
Michael Suha
1241 Bellevue Street
Green Bay, WI 54302
(920) 469-2436

Laboratory Certification Program
General Water Quality\Sludge Testing
Certified parameters from 11-30-2000 to 8-31-2001

Metals

ALUMINUM
BARIUM
CADMIUM
COBALT
LEAD
MERCURY
POTASSIUM
SODIUM
VANADIUM
ANTIMONY
BERYLLIUM
CALCIUM
COPPER
MAGNESIUM
MOLYBDENUM
SELENIUM
THALLIUM
ZINC
ARSENIC
BORON
CHROMIUM
IRON
MANGANESE
NICKEL
SILVER
TITANIUM

Extractable Organics

ACENAPHTHENE
BENZO(A)ANTHRACENE
BENZO(G,H,I)PERYLENE
BIPHENYL
BIS(2-ETHYLHEXYL)PHTHALATE
4-CHLORO-3-METHYLPHENOL
4-CHLOROPHENYL PHENYL ETHER
DIBENZOFURAN
1,4-DICHLOROBENZENE
2,4-DICHLOROPHENOL
DI-N-BUTYL PHTHALATE
2,4-DINITROTOLUENE
FLUORENE
HEXACHLOROCYCLOPENTADIENE
ISOPHORONE
NITROBENZENE
N-NITROSODI-N-PROPYLAMINE
PHENANTHRENE
1,2,4-TRICHLOROBENZENE
ACENAPHTHYLENE
BENZO(A)PYRENE
BENZO(K)FLUORANTHENE
BIS(2-CHLOROETHOXY)METHANE
4-BROMOPHENYLPHENYLETHER
2-CHLORONAPHTHALENE
CHRYSENE
1,2-DICHLOROBENZENE
3,3'-DICHLOROBENZIDINE
2,4-DIMETHYLPHENOL
DI-N-OCTYL PHTHALATE
2,6-DINITROTOLUENE
HEXACHLOROBENZENE
HEXACHLOROETHANE
2-METHYL-4,6-DINITROPHENOL
2-NITROPHENOL
2,2-OXYBIS(1-CHLOROPROPANE)
PHENOL
2,4,5-TRICHLOROPHENOL
ANTHRACENE
BENZO(B)FLUORANTHENE
BENZYL BUTYL PHTHALATE
BIS(2-CHLOROETHYL)ETHER
CARBAZOLE
2-CHLOROPHENOL
DIBENZO(A,H)ANTHRACENE
1,3-DICHLOROBENZENE
DIETHYL PHTHALATE
DIMETHYL PHTHALATE
2,4-DINITROPHENOL
FLUORANTHENE
HEXACHLOROBUTADIENE
IDENO(1,2,3-CD)PYRENE
NAPHTHALENE
4-NITROPHENOL
PENTACHLOROPHENOL
PYRENE
2,4,6-TRICHLOROPHENOL

General Chemistry I

CALCIUM
MAGNESIUM
TURBIDITY
COPPER
MANGANESE
ZINC
HYDROGEN ION (PH)
TOTAL SUSPENDED SOLIDS

Perticides-Herbicides-PCB's

PCB-1016
PCB-1242
PCB-1260
PCB-1221
PCB-1248
PCB-1232
PCB-1254

Purgeable Organics

ACETONE
BENZENE
BROMOFORM
CHLOROBENZENE
CHLOROMETHANE
1,2-DICHLOROBENZENE
DICHLORODIFLUOROMETHANE
1,1-DICHLOROETHENE
CIS-1,3-DICHLOROPROPENE
P-DIOXIANE
METHYLENE CHLORIDE
TETRACHLOROETHENE
1,1,1-TRICHLOROETHANE
TRICHLOROFLUOROMETHANE
ACROLEIN
BROMODICHLOROMETHANE
CARBONTETRACHLORIDE
2-CHLOROETHYL VINYL ETHER
DIBROMODICHLOROMETHANE
1,3-DICHLOROBENZENE
1,1-DICHLOROETHANE
TRANS-1,2-DICHLOROETHENE
TRANS-1,3-DICHLOROPROPENE
EPICHLOROHYDRIN
METHYL ETHYL KETONE
N-TETRADECANE
1,1,2-TRICHLOROETHANE
VINYL CHLORIDE
ACRYLONITRILE
BROMOETHANE
CHLOROETHANE
CHLOROFORM
1,2 DIBROMOETHANE (EDB)
1,4-DICHLOROBENZENE
1,2-DICHLOROETHANE
1,2-DICHLOROPROPANE
DIETHYL ETHER
ETHYLBENZENE
1,1,2,2-TETRACHLOROETHANE
TOLUENE
TRICHLOROETHENE

DATE: 11/30/00
ANTHONY BRIGHT
LABORATORY CERTIFICATION PROGRAM

Each certified laboratory shall provide a copy of this list upon request.

APPENDIX F.

RELEVANT ENCHEM INC. STANDARD OPERATING PROCEDURES

PLEASE RETAIN ALL SOPS IN YOUR COPY EXCEPT FOR THE ONES LISTED IN THE INSTRUCTIONS SHEET. INSERT THE ATTACHED ADDITIONAL SOPS

TITLE: Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by Static Headspace and Gas Chromatography

MATRICES:

This SOP pertains to Aqueous Matrices

DETECTION LIMITS:

The Reporting Limits used in the absence of Project or State Specific Required Detection Limits are recorded in Table A in Appendix B.

The method detection limits were obtained by procedures as outlined in 40CFR Part 136. The spikes were prepared in the same manner as samples. An Estimated Quantitation Limit of 10 ug/L was choosen. This value is above the MDL and within the calibration curve.

1.0 **SCOPE AND APPLICATION:**

1.1 The method outlined within is used for the detection of Methane, Ethane, and Ethylene in Aqueous samples by Static Headspace and Gas Chromatography.

2.0 **SUMMARY OF THE TEST METHOD:**

2.1 Dissolved gases, such as Methane, Ethane, and Ethylene in ground water, are used to determine whether biodegradation of fuels or solvents are occurring in a contaminated aquifer. The ground water samples are heated to 60° C and 1 ml of the static headspace is introduced into a gas chromatograph by a capillary column with a flame ionization detector. The measured response of samples are then compared to an externally calibrated curve for the dissolved gases.

3.0 **DEFINITIONS:**

3.1 Terms used throughout this document are defined in Appendix A.

4.0 **INTERFERENCES:**

4.1 Interferences have not been determined at this time. As with any GC technique, coelution with fuel components and other dissolved gases is always present. Accordingly, it is very important that this analysis is conducted with experienced analysts or under the direct supervision of experienced analysts as to ensure the reliability of the data being generated.

5.0 SAFETY:

5.1 Safety is everyone's business at En Chem, Inc. and everyone is responsible for assisting in reducing unsafe and unhealthy working conditions or potential hazards. The company provides a safe place to work, but employee cooperation is needed to keep it safe. When you see something that does not look safe, or you see someone working in an unsafe manner, inform your supervisor.

5.2 All samples should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique.

5.3 A reference file of material safety data sheets is available to all personnel involved in the chemical analysis.

5.4 Required Safety Equipment is listed in Appendix B Table B

6.0 EQUIPMENT AND SUPPLIES:

6.1 See Appendix B Table C and Table D for a summary.

7.0 REAGENTS AND STANDARDS:

7.1 See Appendix B Table E and Table F for a summary.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE:

8.1 Collection is integral to data quality because of the chemical nature of the compounds of interest. Any headspace in the vials will reduce the recovery of these analytes due to the low molecular masses. Preservative is important to inhibit microorganisms present from releasing these gases after collection biasing results high.

8.2 See Appendix B Table G for a summary.

9.0 **QUALITY CONTROL:**

9.1 All quality control measures shall be subjected to exactly the same preparation procedures as those used on actual samples.

9.2 Quality control measures shall be assessed on an on going basis to determine the usability of the data. The controls measure contamination and statistical measures of accuracy and bias.

9.3 Prior to analysis an Initial Demonstration of Capability shall be performed to validate the method.

9.4 See Table I in Appendix B for the En Chem IDC Summary

9.5 Retention Windows are determined by the laboratory to insure correct identification of target analytes. See Table 1 for the assigned retention time windows. Retention Times are updated where appropriate using the closest CVS to the sample in question.

Table 1
Retention Times

Compound	RT 01	RT 02	RT 03	Expected RT	RT Window	Avg RT	Std Dev
Methane	0.816	0.822	0.826	0.816	0.799-0.832	0.821	0.0005
Etheylene	1.163	1.168	1.174	1.163	1.139-1.186	1.168	0.0005
Ethane	1.437	1.440	1.446	1.437	1.408-1.466	1.441	0.0005

9.6 See Table H in Appendix B for a summary.

10.0 **CALIBRATION AND STANDARDIZATION:**

10.1 Certified Gas Standards are purchased for an approved En Chem supplier at 100 ppm. From these standards an external calibration curve is generated for quantitation.

10.2 Standards must be prepared daily, prior to analysis due to the low molecular masses of the compounds of interest.

10.3 See Table H in Appendix B for a summary.

11.0 **PROCEDURE:**

11.1 Instrument Conditions:

11.2 The chromatographic conditions are listed below in Table 2:

Table 2

Chromatographic Conditions

General	Injector Temperature: Detector Temperature:	200° C 200° C
Column	Carrier Gas (N_2) flow rate: Makeup Gas (N_2) flow rate:	2.0 mL/ Min. 28 mL/min.
Detector	Hydrogen flow rate: Compressed Air flow rate: Split Injection ratio:	30 mL/min. 330 mL/min. 10:1
Temperature Program	Initial Temperature: Program Rate:	40° C 10° C/min to 50° C 70° C/min. to 200° C

11.3 External Instrument Calibration:

11.4 Initial calibration of the instrument is as follows:

11.5 Prepare 5 calibration standards at the following concentration by first dispensing stock gas standard into a tedlar bag.

11.6 Prepare 5 Headspace vials by adding 5 mL of Type II de-ionized water.

11.7 Seal each vial with a Headspace septum and crimp cap using the cap crimping tool.

11.8 Label each vial with the amount of stock gas to be added.

11.9 With the appropriate gastight syringe remove the correct volume of air in the vial and replace with the same volume of the gas standard.

11.10 See Table 3 for standards and amount of gas standard to be added and Tables H and I in Appendix B for a summary.

Table 3

Calibration Table

Amt of Gas Standard (uL)	Methane (ug/L)	Ethane (ug/L)	Ethylene (ug/L)
200	2.8	5.0	5.6
500	7.1	12.5	14
1000	14.2	25	28
2000	28.4	50	56
5000	71	125	140

11.11 Write the file number associated with the standard along with the vial number in the Headspace autosampler into the Analytical Data Log and type the names of the standards in the HP Chem Sequence Log Table and initiate analytical run and temperature program.

11.12 Assess the linearity of the curve using the linear regression calculation provided in Target acquiring software plotted using the concentration in ug/L and Area Response.

11.13 See Table H for acceptance criteria and Table N for Corrective Actions.

11.14 Sample Preparation:

11.15 Samples are prepared by placing a 5 mL aliquot from the 40 mL HCl preserved volatile vials received from sampling into the 10 mL headspace vials using a 5mL pipettor and disposable tip.

11.16 Seal each vial with a Headspace septum and crimp cap using the cap crimping tool.

11.17 Label each vial with correct lab sample identification.

11.18 A Method Blank (MB), Continuing Verification Sample (CVS), Laboratory Control Sample and Duplicate (LCS/D), Matrix Spike and Duplicate (MS/D) are prepared along with the samples to measure the instrument performance

11.19 Blank Preparation:

11.20 Aliquot 5 mL of de-ionized water into a 10 mL Headspace vial using a 5mL pipettor and disposable tip.

11.21 Seal the vial with a Headspace septum and crimp cap using the cap crimping tool.

11.22 Label the vial as the Method Blank

11.23 See Table H in Appendix B for the appropriate frequency of preparation and analysis and Table N for Corrective Actions.

11.24 Calibration Verification Sample Preparation:

11.25 The Calibration Verification Samples are prepared by dispensing 5mL of deionized water into 10mL headspace vials using a 5mL pipettor and disposable tips into the correct number of vials for the size of the analytical run.

11.26 The vials are sealed using a crimping tool to crimp the top over the septum.

11.27 500uL of air is removed from the headspace of the vial using a 500uL gastight syringe.

11.28 500uL of gas standard is removed from the tedlar bag using the 500uL syringe and is immediately injected into the 10mL vial through the septum.

11.29 Label the vials separately as the CVS-1 thru CVS-n.

11.30 See Table H in Appendix B for the appropriate frequency of preparation and analysis and Table N for Corrective Actions.

11.31 LCS Preparation:

11.32 The LCS and Duplicate are prepared by dispensing 5mL of deionized water into a 10mL headspace vial using a 5mL pipettor and disposable tip into two separate 10 mL headspace vials.

11.33 The vials are sealed using a crimping tool to crimp the top over the septum.

11.34 500uL of air is removed from the headspace of the vial using a 500uL gastight syringe.

11.35 500uL of gas standard is removed from the tedlar bag using the 500uL syringe and is immediately injected into the 10mL vial through the septum.

11.36 Label the vials separately as the LCS and LCSD.

11.37 See Table H in Appendix B for the appropriate frequency of preparation and analysis and Table O for Corrective Actions.

11.38 Matrix Spike Preparation:

11.39 The Matrix Spike and Duplicate are prepared by dispensing 5mL of the chosen parent sample using a 5mL pipettor and disposable tip into two separate 10 mL headspace vials.

11.40 The vials are sealed using a crimping tool to crimp the top over the septum.

11.41 500uL of air is removed from the headspace of the vial using a 500uL gastight syringe.

11.42 500uL of gas standard is removed from the tedlar bag using the 500uL syringe and is immediately injected into the 10mL vial through the septum.

11.43 Label the vials separately as the "Parent Lab" MS and "Parent Lab" MSD.

11.44 See Table H in Appendix B for the appropriate frequency of preparation and analysis and Table O for Corrective Actions..

11.45 Analysis:

11.46 Prior to analysis each analytical run, the current calibration must be verified with the analysis of a CVS.

11.47 See Acceptance Criteria in Table H of Appendix B and Table N for Corrective Actions.

11.48 Write the file numbers associated with the samples and quality control samples along with the vial numbers in the Headspace Autosampler into the Analytical Data Log and type the names of the standards in the HP Chem Sequence Log Table and initiate analytical run and temperature program.

11.49 Load the samples into the Headspace Autosampler in the correct order as documented in the Analytical Data Log.

11.50 Samples that have been loaded into the Autosampler require approximately 5 minutes of heating to allow compounds to be driven into the gaseous phase.

11.51 1.0 mL of the gases from the headspace in the vial are then injected onto the column and moved through the column by a flow of nitrogen.

11.52 After the samples have been injected onto the column of the GC the temperature remains at 40°C for one minute.

11.53 The oven temperature is then ramped at 10°C/min. until 50°C is achieved.

11.54 Starting at 50°C the temperature is ramped at a rate of 70°C/min. until 200°C is reached.

11.55 Assess sample data for any potential re-analysis due to exceedance of calibration range.

11.56 Sample dilutions are prepared by dispensing 5mL of deionized water into a 10mL headspace vial using a 5mL pipettor and disposable tip into a 10 mL headspace vials. Dilutions shall produce responses between the midpoint and upper calibration range.

11.57 To produce a dilution, an appropriate amount of DI water is removed from the 10 mL vial using the appropriate gastight syringe and is replaced by an equivalent volume of sample.

11.58 The vials are sealed using a crimping tool to crimp the top over the septum.

11.59 Label the diluted sample vials separately. Repeat analysis starting from 11.45.

12.0 CALCULATIONS:

12.1 Conversions of Gas Standard µmole to ug/L :

x =	Methane	= 16 g /mole	y = Standard contains 100 µmole / g
	Ethane	= 30g / mole	y = Standard contains 105 µmole / g
	Ethene	= 28g / mole	y = Standard contains 101 µmole / g

x g/mole x µmole /1g x 1000 ug/1000 umole x 1 mole/22.4 L = 71 µg/L Methane
= 141 µg/L Ethane
= 126 µg/L Ethene

12.2 Samples results in ug/L are calculated using the linear curve established in the initial calibration.

From linear regression of calibration standard GC responses (RSP) against their known concentrations (AMT in ug/L) derive the following linear equation:

AMT = b + RSP/m1

Using the slope (m1) and the intercept (b) from this equation the concentration of the sample can be calculated from the following equations:

AMTs = (b+RSP/m1)*(D)

Where:

AMTs	=	Concentration of sample in ug/L.
m1	=	slope of the calibration curve
RSP	=	GC response of sample
b	=	intercept of calibration
D	=	dilution factor

13.0 **METHOD PERFORMANCE:**

13.1 The method performance was validated through the Method Detection Limit Study, Initial Demonstration of Capability and Yearly Analyst Demonstrated of Capability. At present Performance Testing Samples are not commercially available.

13.2 See Table I Appendix B for the Initial Demonstration of Capability summary.

14.0 **POLLUTION PREVENTION:**

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order where possible acceptable non-toxic alternative supplies and prepare only those quantities of reagents or standards 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.

14.2 Gas cylinders ordered from Scott Specialty Gases are returned for reconditioning and reuse.

15.0 **DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES:**

15.1 At a minimum there are three levels of data review:

15.2 Analyst/Technician verification at the bench top are looking at instrument performance as it relates to initial calibration, calibration verification, cleanliness.

15.3 Supervisor/Analyst verification after analysis are looking for sample concentration versus linear range of the instrument, typical patterns resulting from the compounds or elements in question, and quality control measure criteria. Data is either accepted without qualification, accepted with qualification, or rejected with samples reprocessed.

15.4 Report Reviewer/Project Management personnel look at the presentation of the data on final reports. The reports are verified for holding time compliance, receipt conditions, method citation and reasonableness, which may include field duplicate analysis, analytical comments and qualifications presented by the previous reviews, and comparing results of similar analytical techniques as it relates to the project site information when available.

15.5 A fourth level of review , data validation is determined from a project or program scope of laboratory services and encompasses the generation Quality Control summaries in the media of "Form Generation."

15.6 See Tables K through M in Appendix B for a summary of the reviews.

16.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1 Assessment of Quality Control measures is done to provide a level of confidence in the data generated. The measures provide documentation that the instrument conditions were reliable during the analysis. See Table K for corrective actions taken in cases where statistical goals were not achieved.

17.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 During analysis, events occur specific to the physical and chemical characteristics of the environmental sample. When possible, with received sample volumes, data generated along with measures that do not meet statistical goals are re-analyzed again to see if the statistical goal can be achieved. When environmental samples do not meet statistical unacceptable data is generated. These events are different from those pertaining to instrument operating conditions. These events occur when the instruments are operating under ideal conditions. See Tables K through O in Appendix B for corrective actions.

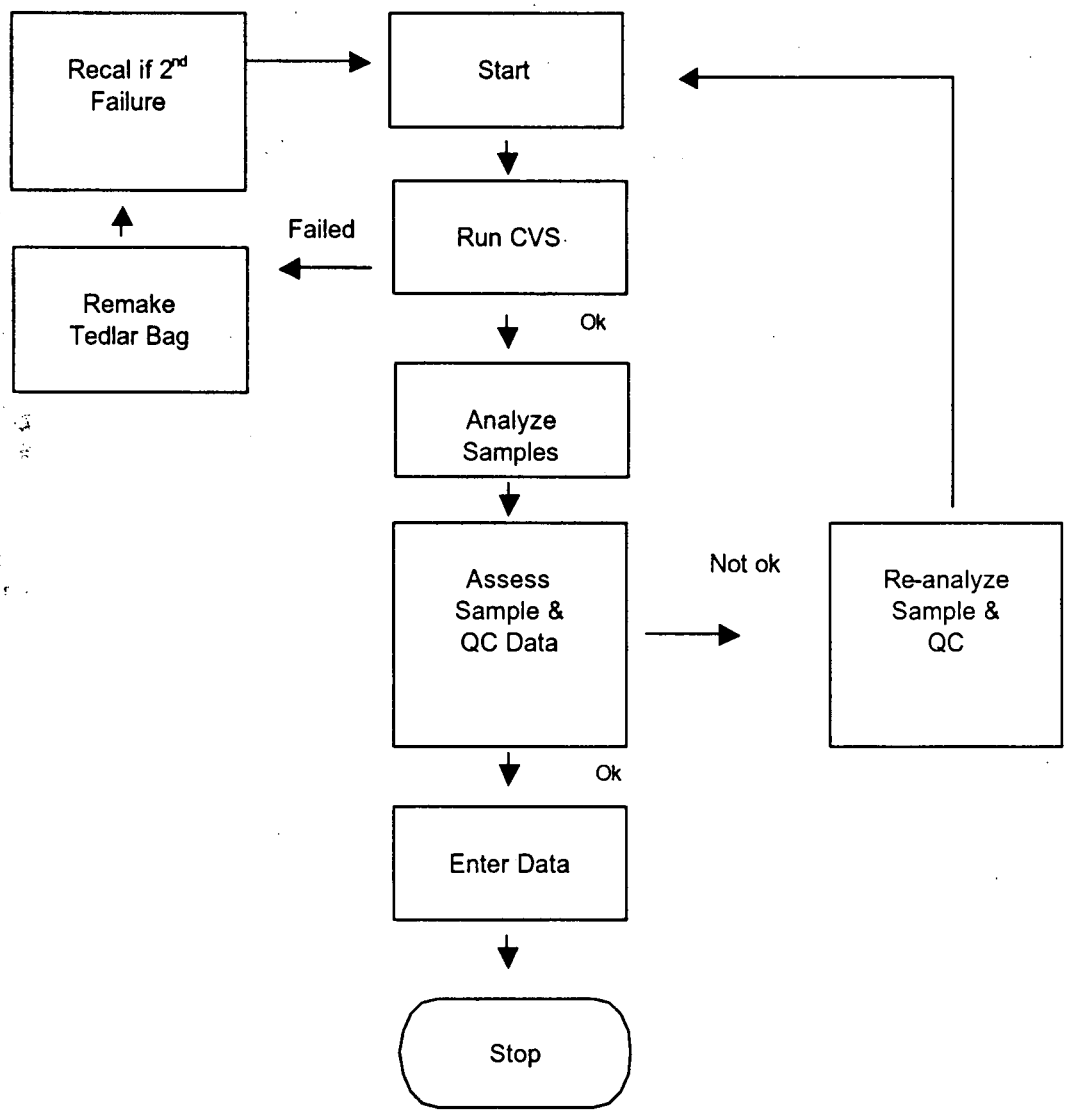
18.0 WASTE MANAGEMENT

18.1 To minimize waste has two benefits. The first benefit is a cost savings to the lab in materials and supplies. The second is a benefit to the environment as less materials need to be disposed.

REFERENCES:

40CFR Part 136
American Society for Quality Control (ASQC), Definitions of Environmental Quality Assurance Terms, 1996
American National Standards Institute (ANSI), Style Manual for Preparation of Proposed American National Standards, Eighth Edition, March 1991
ANSI/ASQC E4, 1994
ANSI N42.23-1995, Measurement and Associated Instrument Quality Assurance for
National Institute of Standards and Technology (NIST)
National Environmental Laboratory Accreditation Conference (NELAC), July 1998 Standards
Random House College Dictionary
US EPA Quality Assurance Division (QAD)
SW846 5021, Update III December 1996
RSK 175

FLOWCHART


Recal if 2nd Failure
Start
Run CVS
Failed
Remake Tedlar Bag
Ok
Analyze Samples
Assess Sample & QC Data
Not ok
Re-analyze Sample & QC
Ok
Enter Data
Stop

VALIDATION DATA:

See Table J in Appendix for a summary of En Chem, Inc. validation of this method.

MANAGEMENT APPROVAL AND REVIEW OF SOPS - POLICY AND DOCUMENTATION

REVIEWED BY: _____ 8/8/2001
Section Supervisor — Date

REVIEWED BY: M. Subra 8-AUG-2001
Quality Assurance Officer — Date

APPROVED BY: _____ 8/8/01
Laboratory Manager — Date

Periodic Review Record

Review Date	24 AUG 01				
Initials	HCS / HKM				

Appendix A
Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)
Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)
Analytical Detection Limit: the smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. (applicable only to radiochemistry)
Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)
Assessment Criteria: the measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)
Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)
Deficiency: an unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)
Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)
Detection Limit: the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence.
Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)
Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)
Equipment Blank: a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)
Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS)
May: denotes permitted action, but not required action. (NELAC)
Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)
Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero

and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)
Must: denotes a requirement that must be met. (Random House College Dictionary)
National Institute of Standards and Technology (NIST): an agency of the US Department of Commerce's Technology Administration that is working with EPA, States, NELAC, and other public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing (PT) to those laboratories testing drinking water and wastewater. (NIST)
Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)
(items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)
Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)
Range: the difference between the minimum and the maximum of a set of values. (EPA-QAD)
Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)
Reagent Blank (method blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)
Record Retention: The systematic collection, indexing and storing of documented information under secure conditions. (EPA-QAD)
Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)
Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)
Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.08)
Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)
Requirement:. denotes a mandatory specification; often designated by the term "shall". (NELAC)
Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)
Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)
Selectivity: (Analytical chemistry) the capability of a test method or instrument to respond to a

target substance or constituent in the presence of non-target substances. (EPA-QAD)
Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)
Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)
Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes. (NELAC)
Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)
Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)
Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)
Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)
Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)
Systems Audit (also Technical Systems Audit): a thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)
Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)
Test Method: an adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)
Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma) (applies to radiobioassay laboratories). (ANSI)
Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 *et seq.*, (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)
Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)
United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural

environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)
Validation: the process of substantiating specified performance criteria. (EPA-QAD)
Verification: confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.
Work Cell: a well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Appendix B

Table A	**Reporting Limits**
Table B	**Required Safety Equipment**
Table C	**Equipment**
Table D	**Supplies**
Table E	**Reagents**
Table F	**Standards**
Table G	**Sample Collection, Preservation, Shipment And Storage**
Table H	**Quality Control**
Table I	**Calibration and Standardization**
Table J	**Initial Demonstration of Capability**
Table K	**Analyst/Technician Data Assessment**
Table L	**Analyst/Supervisor Data Assessment**
Table M	**Report Reviewer/Project Management Data Assessment**
Table N	**Corrective Actions**
Table O	**Contingencies For Handling Out-Of-Control Or Unacceptable Data**

Table A

Reporting Limits

COMPOUND NAME	AQUEOUS (ug/L)	
	MDL	EQL
Methane	2.0	10
Ethane	1.6	10
Ethylene	1.4	10

Table B

REQUIRED SAFETY EQUIPMENT

Item:	Safety Apparel Description	Mandatory for Procedure	Optional
1	Safety Goggles or Glasses		x
2	Lab Coat		x
3	Gloves		x
4	Face Shield		x

Table C

EQUIPMENT

Equipment	Manufacturer	Model(s)	Serial #	Date In Service
GC	Hewlett Packard	5890A	2843A20939	9/96
Autosampler	Hewlett Packard	19395A	274102973	9/96

Table D

Supplies

Supplies	Manufacturer	Vendor	Catalog #
10ul Gastight 1701	Hamilton	Fisher Scientific	14-815-1
25ul Gastight 1702	Hamilton	Fisher Scientific	14-815-29
50ul Gastight 1705	Hamilton	Fisher Scientific	14-824-30
100ul Gastight 1710	Hamilton	Fisher Scientific	13-684-100
250ul Gastight 1725	Hamilton	Fisher Scientific	13-684-102
500ul Gastight 1750	Hamilton	Fisher Scientific	13-684-106
1ml Gastight 1001	Hamilton	Fisher Scientific	14-824-25
5ml Gastight 1005	Hamilton	Fisher Scientific	13-684-96
GASPRO – 0.32 mm id. X 15m	J&W Scientific	VWR Scientific	113-4312
Gold-plated inlet seals	Restek	Restek	21306
Viton O-rings	Restek	Restek	20377
0.4mm Vespel/Graphite ferrules	Restek	Restek	20211
10 mL Headspace Vials	Kimble	Fisher Scientific	20017017
10 mL Headspace Septa	Agilent	Fisher Scientific	58058
Headspace Crimp Tops	Agilent	Fisher Scientific	8759
5 mL Pipettor	Oxford	Fisher Scientific	27-375-13
5 mL Pipet Tips Disposable	Eppendorf	Fisher Scientific	21-371-13
Crimping Tool	West Co., Inc.	—	—
Gas Regulator	Scott Spec Gas,Inc.	Scott, Inc.	5124AV165
Tedlar Bag	Eagle Picher	Fisher Scientific	T20070071UP4M

Table E

Reagents

Reagent	Purity	Concentration	Manufacturer	Vendor	Catalog #
Gas Standard	GC Grade	100 ppm	Scott Specialty Gases	Scott	0504E400380 2
DI Water	Type II	—	—	—	—
Nitrogen	High Purity	—	Michigan Air Gas	MAG	—
Hydrogen	High Purity	—	Michigan Air Gas	MAG	—

Table F

Standards

Standard	Acronym	Concentration	Direction found in Section:	Alias
Scott Gas Standard[1]	—	100 ppm	Section 11.3 of this SOP	Gas standards

1 – Components of Gas Standard is Methane, Ethylene and Ethane

Table G

Sample Collection, Preservation, Shipment And Storage

Matrix	Prep Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
Aqueous	5021 mod	40 mL Glass	HCl	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius

Table H

Quality Control

Preparation Method ⇨ **Quality Control Measure ⇩**	**Mod 5021**
Initial Calibration	Minimum of five levels; lowest level near but above MDL. Correlation Co-efficient > 0.995
Method Blank	One per batch of samples, up to 20 enviromental samples, whichever is more frequent. Acceptable with no detects below Project Specific Requirements. Default below EQLs.
Continuing Calibration Verification	Mid-level calibration standards run every 10 samples and at the beginning and end of each 12 hour time clock. Acceptable if revcovery is 80-120 %
Laboratory Control Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent. Fortify with all target compounds. Acceptable if Recovery is 70-130 % and RPD is < 20%
Matrix Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent. Fortify with all target compounds. Acceptable if Recovery is 70-130 % and RPD is < 20%
Internal Standards	Not Applicable
Surrogate Standards	Not Applicable

Table I

Calibration And Standardization

Analytical Method Acceptance Criteria⇨ Calibration Measure ⇩			**Frequency**	**Accpetance Criteria**
Intitial Calibration			• Prior to initiating analysis when CVS fails acceptance criteria and corrective action of CVS has failed	• Curve is valid if a correlation coefficient of > 0.995 is achieved

Table J

Initial Demonstration

Compound	True Value	REP1 ug/L	REP 2 ug/L	REP3 ug/L	REP 4 ug/L	AVG	AVG REC	STD DEV	Limit %
Methane	7.1	6.268	6.194	6.532	6.162	6.29	88.57	0.167972	70-130
Ethylene	10	11.946	11.954	11.954	11.736	12.08	96.66	0.420461	70-130
Ethane	12	13.227	12.938	12.938	12.137	12.75	91.05	0.462943	70-130

Table K
Analyst/Technician Data Assessment

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**If these conditions are not achieved ⇒**
Initial Calibration	• 1
Initial Calibration Verification	• 1
Calibration Blank	• 1
Method Blank	• 2
Internal Standard Area	• 3
Continuing Calibration Verifications	• 4
Surrogate Recovery	• 5
Accuracy & Precision Laboratory Control Spikes	• 6
Accuracy & Precision Matrix Spikes	• 7

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration. No data can be reported. Generate on Non-Conformance Memo.

2. In the absence of project specific requirements, sample detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified. Generate on Non-Conformance Memo.

3. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. Re analyze sample if volume allows. Generate on Non-Conformance Memo.

4. In the absence of project specific requirements, immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV. Generate on Non-Conformance Memo.

5. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. If the program allows report the concentrations noting the surrogate recovery is most likely due to co-elution with target compounds. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. Re analyze sample if volume allows. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the in-house QC limits, and no further action is required. If, upon re-analysis the recovery is again not within limits, report the data as an "estimated concentration." If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user. Generate on Non-Conformance Memo.

6. In the absence of project specific requirements, the LCS and LCSD are spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If the LCS and LCSD recoveries do not meet those limits as required in the method or program, the samples shall be re-analyzed. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged to reflect the deficiencies. Generate on Non-Conformance Memo.

7. In the absence of project specific requirements, when the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If sample volume allows re-analysis, it shall be performed to verify the matrix effect. When sample volume does not re-analysis, the parent sample and samples from that project site shall be flagged to reflect the deficiencies. Generate a Non- Conformance Memo.

Table L
Analyst/Supervisor Data Assessment

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇓	If Criteria in Table H are not achieved ⇒
Initial Calibration	• 1
Initial Calibration Verification	• 1
Calibration Blank	• 1
Method Blank	• 2
Internal Standard Area	• 3
Continuing Calibration Verifications	• 4
Surrogate Recovery	• 5
Accuracy & Precision Laboratory Control Spikes	• 6
Accuracy & Precision Matrix Spikes	• 7

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration. No data can be reported. Generate on Non-Conformance Memo.

2. In the absence of project specific requirements, sample detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified. Generate on Non-Conformance Memo.

3. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. Re analyze sample if volume allows. Generate on Non-Conformance Memo.

4. In the absence of project specific requirements, immediately after the flawed CVS analyze another CVS or re-analyzed all samples set after the last acceptable CVS. Generate on Non-Conformance Memo.

5. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. If the program allows report the concentrations noting the surrogate recovery is most likely due to co-elution with target compounds. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. Re analyze sample if volume allows. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the in-house QC limits, and no further action is required. If, upon re-analysis the recovery is again not within limits, report the data as an "estimated concentration." If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user. Generate on Non-Conformance Memo.

6. In the absence of project specific requirements, the LCS and LCSD are spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If the LCS and LCSD recoveries do not meet those limits as required in the method or program, the samples shall be re-analyzed. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged to reflect the deficiencies. Generate on Non-Conformance Memo.

7. In the absence of project specific requirements, when the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If sample volume allows re-analysis, it shall be performed to verify the matrix effect. When sample volume does not re-analysis, the parent sample and samples from that project site shall be flagged to reflect the deficiencies. Generate a Non- Conformance Memo.

Table M
Report Reviewer/Project Management Data Assessment

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**If Criteria in Table H are not achieved ⇒**
Holding Time Compliance	• 1
Sample Receipt Conditions and Preservation	• 2
Method Citation	• 3
Reasonableness: Field Duplicate	• 4
Reasonableness: Analytical Comments Qualifications	• 5

1. Flag results with an H and the number of days past hold in parenthesis e.g. H(5).

2. In prose describe the receipt conditions as they relate to the acceptance criteria list in Table G

3. Compare to regulatory program in QAPjP or Chain of Custody.

4. Are duplicate with 40% agreement. If not, internally flag for review.

5. Do related suites of tests agree or are reasonable.

Table N
Corrective Actions

Analytical Method Acceptance Criteria⇨ Quality Control Measure ⇩	**Method Citation SW If these conditions are not achieved ⇒**	**Corrective Action (Key below)**
Initial Calibration	• Criteria as listed in Table H	• 1
Method Blank	• Criteria as listed in Table H	• 2
Continuing Calibration Sample	• Criteria as listed in Table H	• 3

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration

2. Samples detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified.

3. Immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV.

Table O
Contingencies For Handling Out-Of-Control Or Unacceptable Data

Analytical Method Acceptance Criteria⇨ Quality Control Measure ⇩	**If these conditions are not achieved ⇒**	**Corrective Action (Key below)**
CVS	• Criteria as listed in Table H	• 1
Lab Control Spike and Duplicate	• Criteria as listed in Table H	• 2
Matrix Spike and Duplicate	• Criteria as listed in Table H	• 2

1. If CVS does not meet acceptance criteria abort analytical run and discard gas standard in tedlar bag. Fill new tedlar bag with a fresh gas standard and re-prep spiked samples and Duplicates. Re-start analytical run with a new CVS and replaced spiked samples.

2. If spiked samples do not meet acceptance criteria abort analytical run and discard gas standard in tedlar bag. Fill new tedlar bag with a fresh gas standard and re-prep spiked samples and Duplicates. Re-start analytical run with a new CVS and replaced spiked samples.

ANALYTICAL METHOD

TITLE: Ion Chromatography

DEPARTMENT: Inorganic – Wet Chemistry

APPLICATION: Anions in drinking and surface waters, and groundwater.

PROCEDURE SUMMARY:

Method is used to determine Bromide, Fluoride, Chloride, Nitrite, Nitrate, Ortho-phosphate and Sulfate in ground water and surface water by use of an Ion Chromatograph.

REPORTING LIMIT:

See Attachment 1.

APPROVED BY: _____ 1-29-01
Jeffrey A. Gordon — Date
Inorganic Section Supervisor

_____ 1-29-01
Gregory J. Graf — Date
Quality Assurance Officer

_____ 1/31/01
Glen A. Coder — Date
Laboratory Manager

Annual Review

Date:					
Initials:					

SAMPLE HANDLING AND PRESERVATION:

Sample can be collected in glass or plastic. Samples need to be unpreserved. Sample holding times: NO2, NO3, OPO4: 48 hours; F, CL, SO4: 28 days; Br: 28 days.

INTERFERENCES:

Samples that contain particles larger than 0.45 microns and reagents solutions that contain particles larger than 0.20 microns require filtration to prevent damage to the instrument column or flow system.

APPARATUS AND MATERIALS:

DX – 120 Ion Chromatograph
5.0 mL sample vials and filter caps
Volumetric flasks: 100 mL, 1000 mL
Pipettors: adjustable and fixed volume (3.0, 1.5, 1.0, 0.10, 0.050, 0.010 mL)

REAGENTS AND STANDARDS:

NOTE: All solutions must be made with Milli-Q® water.

Prepare Eluent:

Dilute 10 mL of concentrated eluent to 1 L with Milli-Q® water.

Prepare Anion Calibration Standards:

Dilute 3.0, 2.5, 1.5, 1.0, 0.10, and 0.020 mL of 1000 ppm APG Standard of each of the following anions (Br, F, CL, NO_2, NO_3, OPO_4, SO_4) in 100 mL volumetric with Milli-Q® water to make 30, 25, 15, 10, 1.0, 0.20 ppm standard solutions. Prepare standards weekly, except those < 1 ppm which are prepared daily.

Prepare Anion Check Standard:

Dilute 1.5 mL of 1000 ppm APG Standard of each of the following anions (Br, F, CL, NO_2, NO_3, OPO_4, SO_4) in 100 mL volumetric with Milli-Q® water to make a 15 ppm standard. Source of standard should be of a different lot than that of calibration standards. Prepare weekly.

PROCEDURE:

1. Execute PeakNet.
2. Click on "Run Method".
3. Open method file: "Startup".

4. Run manual baseline until stable, usually 10 minutes.

5. Open "Schedule" from PeakNet main menu.

6. Type in analytical run you wish to perform:

    a. For calibration standards, be sure to include sample type as "calibration std" and level as: Cal30.0= level 1, Cal25.0 = level 2, Cal15.0 = level 3, Cal10.0 = level 4, Cal1.0 = level 5, Cal0.2 = level 6, Cal0.0 = level 7.

    b. ICV, ICB, CCV, CCB: Sample type is "check std". CCV, ICV = level 1 and CCB, ICB = level 2.

    c. All others need sample type "sample".

    d. Enter Method: En Chem Anions.met (Br, CL, F, NO_2, NO_3, OPO_4, SO_4) for IC "A", or EnChem Anions B for IC "B".

    e. Enter data file: yymmdd A or B.

    f. Save schedule: yymmdd A or B.

    g. After last CCB enter sample "shutdown" with Method: shutdown.met.

7. From run window: Load schedule:

    a. Click on mode tab.

    b. Click on "run via external signal".

8. Load autosampler.

    a. Pour 5 mL of sample into each vial.

    b. Seat filter cap so top of cap is even with top of vial.

9. Press run/hold button of autosampler.

10. Once analytical run has completed, go to PeakNet main menu and open "Optimize".

11. Each sample produces it's own .dat file and chromatograph. Open each file in the optimize window, check and correct peak naming and void volumes. Excess peaks can be deleted.

12. Once all samples have been optimized, open "Batch" from PeakNet main menu.

    a. Select input:

        1. Select schedule which you wish to batch.

        2. Lines used should be one less than total lines in schedule.

b. Click output tab:

1. Click on summary report

c. Click export tab:

1. Type in name of download file: e.g.: G:\data\inorgani\ic\yymmdd A or B.

CALIBRATION AND STANDARDIZATION:

Perform calibration with standards at the following levels (ppm): 0.20, 1.0, 10, 15, 25 and 30. These standards define the linear range for the analysis. The correlation coefficient must be 0.995 or greater.

QUALITY CONTROL:

Correlation Coefficient (r-value)
The correlation coefficient, the measure of linearity of a standard curve, must be 0.995 or greater. If the value is less than 0.995, recalibrate the instrument.

Initial Calibration Verification (ICV)
The ICV must be analyzed immediately after calibration and meet the rejection criteria of ± 10% of the true value. Recalibrate if the ICV fails. The concentration of the ICV should be near the mid-point of the calibration curve.

APG Check Standard
An APG check standard for each anion must be analyzed weekly to check column efficiency, accuracy, etc. Rejection criteria is ± 10% of the true value. If the APG fails, the separation column will be replaced or taken out of service until it can be cleaned. Results will be recorded in the IC Daily Log.

Initial Calibration Blank (ICB)
The ICB must be analyzed after the ICV. The absolute value must be ≤ EQL. Recalibrate if it fails.

Continuing Calibration Verification (CCV)
The CCV is analyzed after every 10 samples. Rejection criteria is ± 10% of true value. If the CCV fails, the problem must be corrected and the previous 10 samples between the CCV and last CCB must be reanalyzed. Concentration of the CCV should be near the mid-point of the calibration curve. As long as the CCVs that bracket the samples to be reported for the analytes of interest are within the acceptable limit, the run is acceptable.

Continuing Calibration Blank (CCB)
The CCB is analyzed after every CCV. The absolute value must be ≤ EQL. If the CCB fails, the problem must be corrected and the previous 10 samples between the last CCB and the CCV must be reanalyzed.

Laboratory Control Sample (LCS)
The LCS is carried through all preparation procedures and analyzed for each matrix type with a frequency of 5%. See Attachment 1 for control ranges. In cases where the LCS is outside of acceptable ranges all samples prepared in that batch must be reprepared and reanalyzed.

Method Blank (MB)
The MB is carried through all prep procedures and analyzed with a frequency of 5%. Rejection criteria is >EQL. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

ACCURACY

One matrix spike and matrix spike duplicate are analyzed for each group of samples that are similar in matrix at a frequency of 5%. Both QC samples must be calculated for accuracy. See Attachment 1 for control range.

$$\text{Spike Percent Recovery} = \frac{SSR - SR}{SA}$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

If both spike recoveries are outside of the specified control limit, the corresponding parent sample is to be diluted and MS/MSD performed.

Dilute appropriately until an acceptable recovery is obtained. If only the matrix spike OR the matrix spike duplicate is out of control for accuracy, then the corresponding parent sample is flagged with the [MS qualifier.

If there is insufficient sample volume to perform a matrix spike and a matrix spike duplicate, an LCS and an LCS DUP must be used in its place.

PRECISION

Matrix spike duplicate samples are analyzed 1 per batch or at a frequency of 5%, for samples that are similar in matrix.

For matrix spike duplicate samples, relative percent difference (RPD) is used to calculate compliance. See Attachment 1 for control limits.

Calculation:

$$RPD = \frac{|MS - MSD|}{(MS+MSD)/2} \times 100$$

MS = Method Spike Value
MSD = Method Spike Duplicate Value

If the RPD is outside of the acceptable control limits, the reported sample result is to be qualified with an [* flag.

Sample Result Calculations:

Aqueous Sample Calculation:

Raw Data result (mg/L) X DF = Final Result (mg/L)

DF = Dilution Factor

SAFETY:

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

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

ANION	REPORTING Limit (mg/L)	LCS Control Limit (% REC)	MS/MSD Control Limit (% REC)	RPD Control Limit (MAX.%)
Bromide	0.20	90-110	90-114	5
Fluoride	0.10	90-117	76-122	4
Chloride	2.0	88-111	76-113	7
Nitrate	0.20	90-110	90-110	4
Nitrite	0.20	90-110	90-110	4
Ortho-phosphorus	1.0	79-129	75-145	5

** Control limits are updated periodically. The control limits that are in use at the time of analysis will be used and made available to data validators.

En Chem, Inc.

Quality Assurance Document

SET No: 1

STANDARD OPERATING PROCEDURE

TITLE: Nitrate and Nitrite, Automated Analysis

DEPARTMENT: Inorganic - Wet Chemistry

APPLICATION: Drinking, surface, and ground water, domestic and industrial wastewaters and solubilized solid matricies.

REFERENCES: EPA 600 4-79-020, Revised March 1983, Method 353.2
Lachat QuikChem Method No. 10-107-04-1-C

PROCEDURE SUMMARY:

Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl) ethylenediamine dihydrochloride. The resulting water soluble dye has a magenta color which is read at 520 nm. Nitrite can be determined by removing the cadmium column.

APPROVED BY: Jeffrey A. Gordon — Date: 1-31-01
Jeffrey A. Gordon
Inorganic Section Supervisor

Gregory J. Graf — Date: 1/31/01
Greg J. Graf
Quality Assurance Officer

Glen A. Coder — Date: 1/31/01
Glen A. Coder
Laboratory Manager

Annual Review

Date:					
Initials:					

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SAMPLING HANDLING AND PRESERVATION:

Total N (NO_3+NO_2): Collect 250 mL of sample in a plastic or glass container, preserve to a pH <2 with sulfuric acid, and refrigerate to 4°C. Holding time is 28 days from date of collection.

For nitrite (NO_2) or nitrate (NO_3): Collect 250 mL of sample in a plastic or glass container and refrigerate to 4°C. Holding time is 48 hours from time of collection.

Drinking Water samples to be analyzed for nitrate: If not able to analyze within 48 hours, preservation with sulfuric acid to pH <2 at the time of collection and analysis within 14 days. Acid preservation must be done within 15 minutes of sample collection. N(NO_3+NO_2) is reported rather than NO_3. Drinking Water samples from a chlorinated water supply do not require cooling or preservation and can be analyzed within 14 days.

SAFETY:

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

INTERFERENCES:

Build up of suspended matter in the reduction column will restrict sample flow. Since nitrate-nitrogen is soluble, the sample may be prefiltered.

Low results may be obtained for samples that contain high concentrations of iron, copper, or other metals. EDTA is added to the buffer to reduce this interference.

Residual chlorine can interfere by oxidizing the cadmium column.

Samples that contain oil and grease coat the surface of the cadmium in the column. A pre-extraction with an organic solvent will eliminate this interference.

APPARATUS AND MATERIALS:

Lachat QuikChem Automated Ion Analyzer
520 nm interference filter
17 cm sample loop
10-107-04-1-C nitrate + nitrite, nitrite manifold
Volumetric flasks: 100 mL, 1000 mL
Pipettor: adjustable
Graduated cylinder: 100 mL
Cadmium-Copper Reduction Column (Lachat Part #50237)

REAGENTS:

Deionized (D.I.) water
Copper sulfate ($CuSO_4 \cdot 5\ H_2O$)
Disodium ethylenedinitrilotetraacetic acid (EDTA disodium salt)
Ammonium hydroxide (NH_4OH)
Hydrochloric acid (HCl), concentrated
Phosphoric acid (H_3PO_4), concentrated
Sulfanilamide
N-1-naphthylethelenediamine dihydro chloride
Potassium nitrate (KNO_3), 2 sources
Potassium nitrite (KNO_2), 2 sources
Chloroform
Helium gas

Prepare Ammonium Chloride, Buffer, pH 8.5

- In the hood, in a 1 liter volumetric flask add 500 mL D.I. water, 105 mL concentrated hydrochloric acid (HCL), 95 mL ammonium hydroxide (NH_4OH) and 1.0 gram disodium EDTA.
- Dissolve and dilute to the mark. Mix well.
- Prepare fresh weekly.

Prepare sulfanilamide color reagent as follows:

- To approximately 800 mL D.I. water, add 100 mL of 85% phosphoric acid (H_3PO_4), 40 grams sulfanilamide, and 1.0 gram N-1-naphthylethelenediamine dihydro chloride.
- Shake to wet, and stir to dissolve for 30 minutes. Dilute to the mark in a 1 liter volumetric flask and invert to mix . Store at 4°C. Shelf Life is 1 year.

Carrier is degassed D.I.

Degas ammonium chloride and sulfanilamide reagents before use with helium gas for 2 minutes per liter of solution.

Prepare 1M hydrochloric acid, HCl, as follows:

- Add 8 mL conc. HCl to 92 mL D.I. water in a 100 mL volumetric flask.
- Mix well. Shelf life is 1 year.

Prepare copper sulfate solution, 2%, as follows:

- Dissolve 20 grams $CuS0_4 \cdot 5\ H_20$ in a 1 L volumetric flask of D.I. water.

- Mix well. Shelf life is 1 year.

Prepare stock nitrate standard, 100 mg/L, as follows:

- Dry potassium nitrate in an oven at 105° C for 24 hours.
- Dissolve 0.722g dried potassium nitrate ($KN0_3$) in D.I. water and dilute to 1 liter in a volumetric flask.
- Preserve with 2 mL chloroform, mix well, and refrigerate. Standard is stable for 6 months.

Prepare stock nitrite standard, 100 mg/L, as follows:

- Dry potassium nitrite in an over at 105° C for 24 hours.
- Dissolve 0.607g dried potassium nitrite (KNO_2) in D.I. water and dilute to 1 liter in a volumetric flask.
- Preserve with 2 mL chloroform and refrigerate. Standard is stable for 6 months.

Prepare one stock standard from one source for calibration standards and one stock standard for calibration verifications from another source. (ALL should be prepared DAILY)

Prepare calibration standards for nitrate as follows:

- Pipette the indicated amounts of stock nitrate standard into a 100 mL volumetric flask and dilute to volume with D.I. water.

mL Stock Standard/100 mL	NO_3,mg/L
0.10	0.10
0.20	0.20
0.30	0.30
0.50	0.50
1.0	1.00
2.5	2.50
3.0	3.00

Prepare CCV standard for nitrate as follows:

1.0	1.00

NOTE: 1.0 mg/L nitrite and 1.0 mg/L nitrate standards are needed for measuring the efficiency of the cadmium column.

Prepare calibration standards for nitrite, as follows:

- Pipette the indicated amounts of stock nitrite standard into a 100 mL volumetric flask and dilute to volume with D.I. water.

mL Stock Standard/100 mL	NO_2, mg/L
0.05	0. 05
0.10	0.10
0.30	0.30
0.50	0.50
1.0	1.00
2.0	2.00

Prepare standards for CCV for nitrite:

CCV	0.50	0.50

Order repacked cadmium column from LACHAT or, if necessary, prepare cadmium-copper reduction column as follows:

Cadmium Preparation

1. Place 10 - 20 grams coarse cadmium granules into a 250 mL beaker and wash with 50 mL acetone, 50 mL D.I. water, and two 50 mL portions of 1 M HCl. If using cadmium for the second time, rinse with 1 M HCl before beginning process.

2. Add 100 mL of 2% copper sulfate solution to the cadmium prepared in Step 1. Swirl gently for about 5 minutes. Decant the liquid and repeat with a fresh 100 mL portion of copper sulfate. Continue this process until colloidal copper is visible in the supernatant (a red-brown color). Decant and wash with at least ten 100 mL portions of D.I. water to remove the colloidal copper. The cadmium should be black. The cadmium granules may be stored in a stoppered bottle under the ammonium chloride carrier solution.

Packing the Column

1. Into a 15 cm length of 4 mm ID glass tube, place a small piece of polyurethane foam loosely in the end.

2. Wrap the threading of the tube with tygon tape.

3. Insert the plugged end of the glass tube into the tygon sleeve of the column connector fitting until the end of the column connector butts onto the glass tube.

4. Insert the Teflon tube in the end fitting and fill the whole tube with the ammonium chloride carrier solution, taking care that no air bubbles are introduced.

5. Place the copperized cadmium granules in the column with the aid of a funnel. Tap the column gently every 12 cm to pack the granules.

6. When the column is packed to within ~ 2 mm of the end of the glass tube, insert another foam plug, wrap threading with tygon tape, and attach the column end fitting.

7. Store the column with the ends connected. Air pockets or allowing the column to dry out will necessitate repacking.

Cadmium Column Insertion Procedure:

1. Before inserting the column, pump all reagents into the manifold. Turn the pump off. On the column, disconnect the center tubing from one of the union connectors and immediately connect to the outlet tubing of the buffer mixing coil.

2. Connect the open tubing on the column to the tee fitting where the color reagent is added. DO NOT let air enter the column. (If air enters the column, efficiency will decrease).

3. Return pump to normal speed. The direction of reagent flow through the column is not relevant. If air is accidentally introduced into column, turn the pump on maximum, and tap firmly with a screwdriver handle, working up the column until all air is removed.

PROCEDURE:

1. Set up instrument. (See En Chem Method WCM-29)

2. Connect 10-107-04-1-C manifold, with 2 state switching valve to place cadmium column in-line with the manifold.

3. Install 520 nm interference filter.

4. Install 17 cm sample loop.

5. Calibrate instrument with the appropriate set of standards beginning with the highest standard first. NOTE: All analyses on the LACHAT are performed using one replicate.

6. Measure the column efficiency (see Appendix A).

7. For Solid Matricies:
Weigh 10 grams of the material into a beaker. Add 100 mL of D.I. water. Stir the mixture for 1 hour. Filter the slurry and analyze the filtrate as a water matrix. The result should be reported as "soluble" .

Calibration

The instrument must be calibrated every time it is set up. The correlation coefficient must be 0.995 or greater. If not recalibrate.

Quality Control

Initial Calibration Verification (ICV)
The ICV must be analyzed immediately after calibration and meet the rejection criteria of ±10%. Recalibrate if it fails.

Initial Calibration Blank (ICB)
The ICB must be analyzed after the ICV. The absolute value must be less than the absolute value of the estimated quantitation limit (EQL). If not, recalibrate.

Laboratory Control Sample (LCS)
Prepare a reference sample which is purchased from Analytical Products Group, Inc. (APG) as a setpoint standard. This standard must be analyzed for each 20 samples and meet current control limits.

Method Blank
The MB is carried through all prep procedures and analyzed with a frequency of 5%. Rejection criteria is < LOD. Other criteria may apply, such as regulatory limit and analyte concentration in samples.

Column efficiency
Rejection criteria is ±15%. Insert new column with fresh cadmium, if it fails.

Continuing Calibration Verification (CCV)
The CCV is analyzed after every 10 samples. The rejection criteria is ±10%. If it fails, recalibrate and repeat the previous 10 analyses.

Continuing Calibration Blank (CCB)
The CCB is analyzed after every CCV. The absolute value must be less than the absolute value of the EQL. If not, recalibrate and repeat the previous 10 analyses.

Matrix Spike
A spike must be performed on each group of samples of a similar matrix type with a frequency of 5%. Recovery must meet the current control limits.

Matrix Spike Duplicate
A matrix spike duplicate must be analyzed on each group of samples of a similar matrix type with a frequency of 5%. Recovery must meet the current control limits for accuracy. The difference between the MS and MSD must meet current control limits for precision.

Calculations

Column efficiency - See Appendix A

Samples:

The instrument provides calculated sample results in mg/L, calculations are only necessary if a dilution was used.

Raw Data Value (mg/L) x Dilution Factor = NO3 + NO2 (mg/L)

Accuracy:

A matrix spike and matrix spike duplicate must be performed on each group of samples of a similar matrix type with a frequency of 5% and meet the current control limits for accuracy.

Spike calculation:

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

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

If there is insufficient volume available for an MS/MSD, perform an LCS/LCSDUP.

Precision:

A matrix spike duplicate must be analyzed on each group of samples of a similar matrix type with a frequency of 5% and meet the current control limits for precision.

Relative percent difference (RPD) calculation:

$$RPD = \frac{|MS-MSD|}{(MS+MSD)/2} \times 100$$

MS = Matrix Spike Value
MSD = Matrix Spike Duplicate Value

If there is insufficient volume available for an MS/MSD, perform an LCS/LCSDUP

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.

Appendix A

zellweger analytics

Lachat Instruments Division
Service Business Unit
6645 West Mill Road
Milwaukee, WI 53218-1239 USA
Tel: (1) 414 358 4217
Fax: (1) 414 358 4206
Email: lachat_support@zelana.com

Column Inspection

- Visually inspect the column. Check for air bubbles in the column or lines, gaps in the column or any change in the cadmium surface characteristics. Cadmium granules should be dark gray.
- If air bubbles are present in column because of operator error, turn the switching valve in-line. Turn the pump speed to maximum. Tilt the column to an almost vertical position (outlet should be at the top) and tap firmly with a screwdriver handle, being careful not to break the column. If air cannot be removed, the column should be repacked. Cadmium columns should be stored filled with buffer solution. If air enters the column, efficiency will decrease.
- Check the flow efficiency by disconnecting the cadmium column from the manifold and reconnecting to a green-green pump tube. Pump buffer through the packed column and collect in a graduated cylinder. The flow rate with the column connected should be greater than 4.0 mL/min.

Column Efficiency

The column efficiency can be determined as follows:

- Calibrate with NO_3 standards.
- Run a know concentration NO_2 standard
- Run a matching concentration NO_3 standard
- The column efficiency is determined by the equation:

$$E = \frac{[NO_3]}{[NO_2]} \times 100$$

where:

E = column efficiency

$[NO_3]$ = concentration of nitrate standard

$[NO_2]$ = concentration of nitrite standard

- If the efficiency is less than 90%, the column should be repacked.

New and Repacked Cd columns from Zellweger-Lachat are guaranteed to have an efficiency of 90% or higher. The columns are individually inspected to ensure no air has been introduced at the factory.

If you need further assistance, please call the
Technical Support Department @ 414-358-4217

En Chem, Inc.

Quality Assurance Document

STANDARD OPERATING PROCEDURE

TITLE: Total Organic Carbon Analysis

DEPARTMENT: Inorganic - Wet Chemistry

APPLICATION: Determination of organic carbon in soil, sludge and solid waste.

REFERENCE: EPA Manual SW846, 3rd Edition, Method 9060 Modified
Dohrmann DC-190 High Temperature TOC Analyzer Operating Manual, September 1994 Revision C

PROCEDURE SUMMARY:

Inorganic carbon from carbonates and bicarbonates is removed by acid treatment. The sample is vaporized and swept by the continuous oxygen flow to the 800 °C combustion zone where all carbonaceous matter is oxidized to CO_2. The carbon dioxide is bubbled through an acidified liquid and then routed through a mist trap which together serve to remove any entrained water and scrub out any corrosive species formed. Finally, the gas is swept to the linearized non-dispersive infrared detector which is made specific for CO_2 measurement.

LABORATORY DETECTION AND REPORTING LIMITS:

	LOD	EQL
Soil:	91 mg/kg	500 mg/kg

Approximate working range of method: LOD to 30,000 mg/kg.

APPROVED BY: _____ 8/3/01
Michael J. Heimann — Date
Wet Chemistry Group Leader

_____ _____
Julie A. Trivedi — Date
Quality Assurance Officer

_____ _____
Glen A. Coder — Date
Laboratory Manager

Annual Review

Date:					
Initials:					

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SAMPLE HANDLING & PRESERVATION:

The sample is collected in a glass jar and refrigerated at 4°C. The recommended holding time is 28 days from sample collection.

SAFETY:

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

INTERFERENCES:

Carbonate and bicarbonate carbon represent an interference under the terms of this test and must be removed or accounted for in the final calculation. Removal of carbonate and bicarbonate by acidification can result in the loss of volatile organic substances resulting in a low bias.

Bacterial decomposition and volatilization of the organic compounds are minimized by maintaining the sample at 4°C and analyzing within the specified holding time.

APPARATUS:

Dohrmann Model 183 Total Organic Carbon Analyzer or equivalent
Syringes: 100 µL
Volumetric flasks: 100 mL
Forceps
Watch glass
Small spatula
Platinum boat
Crucibles
Magnetic stir bars
Magnetic stir/hot plate
Aluminum dishes
Spatulas
Balances: top loader, analytical capable of 0.1 mg resolution and accuracy

REAGENTS:

Milli-Q deionized water
Potassium hydrogen phthalate (KHP), primary standard grade
Sulfuric acid (H_2SO_4)

Prepare potassium hydrogen phthalate, 2000 ppm carbon, as follows:
Dissolve 425 mg KHP in 100 mL Milli-Q water.
Store in an amber bottle and refrigerate. Shelf life is one month.

NOTE: Another 2000 ppm standard is prepared from a second source that is used as the ICV/CCV. Prepare calibration standards daily.

Prepare sulfuric acid solution, 5% (v/v), as follows:
Dilute 10 mL of concentrated H_2SO_4 to 200 mL with Milli-Q water, slowly adding the acid. Mix well and place in a glass bottle. Shelf life is 1 year.

Prepare Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB) and Preparation Blank (Method Blank):
Use the same D.I. water that was used for the preparation of the standards. Inject 40 µL onto an ottawa sand (40 mg) which has gone through the same sample preparation procedures.

Prepare Laboratory Control Sample (LCS):
Use potassium hydrogen phthalate stock standard with a concentration of 2000 ppm. Inject 19 µL (results in a 950 mg/L spike for the LCS) into the boat.

PROCEDURE:

A. Sample Preparation

1. Place a representative homogeneous sample, at least 10 grams, in a crucible. Add a stir bar.

2. Place on a stirring hot plate. Slowly add 5% H_2SO_4, dropwise, until sample is saturated and stir bar turns freely.

NOTE: Sample will effervesce in the presence of inorganic carbon.

3. Set temperature at ~ 70°C to help drive the reaction.

NOTE: Process a blank using Ottawa sand for each 20 samples prepared.

4. Check sample pH occasionally. pH should be maintained at <2. If pH is >2, add more acid.

5. Continue to heat, stir, and add acid until effervescence is no longer visible or no longer than 24 hours.

NOTE: This can be difficult to determine in some samples. Check by removing from stirring hot plate and allow to sit for several minutes. Swirl sample and look for air bubbles.

6. Dry in an oven at 103-105° overnight.

B. Sample Analysis

1. Set up instrument. See EN CHEM SOP WCM-18.

2. The boat is introduced into the furnace and allowed to "bake-out".

3. Refer to SOP EN CHEM WCM-18 for set up of Soils/Boat.
   a. Set instrument to NPOC/BOA mode. After the furnace has warmed up to the set temperature, have a prepared boat ready to inject. Place the boat

in the slide. Close the lid. Press start. Type in the mass of the of the sample. Hit Enter. Wait until the inject light comes on.

b. When the unit chimes, inject/slide the sample/standard. It will chime again when it is finished. Enter Y or N for replicate. Print the analysis if N.

c. Continue on to the next sample or standard.

Calibration

4. Calibrate the instrument as follows:

a. Using a 2000 ppm KHP standard, inject 40 µL of the standard into the boat by lifting the cover to the sample port.

b. Close the cover, hit start and enter the weight as 40 mg. Hit Enter. Wait for chime, then push sample in via the magnet.

c. Instrument will chime when analysis is complete. Pull magnet back.

d. Allow sample boat to cool a minute or two.

e. Repeat standard injection two more times.

f. When three calibration standards are read, Press NO to continue. The instrument will calculate a mean and standard deviation. To update the calibration factor, push the calibration button. Find and select the Update Calibration Factor (#5). Press #5 and the unit will scroll to it and update. Write this factor down. Continue to calibrate with the next standard.

Analysis QC

5.

a. Analyze a 500, 1000, 2000 and 3500 ppm standards from the same source as the calibration. The % recovery for each standard must be ± 10% of the true value. Then analyze an ICV sample, which must be ± 10%.

b. The ICB value should be less than the detect value The ICB must be analyzed after the ICV. The absolute value must be less than the (EQL).

c. LCS: Weigh 10 mg of the dried ottawa sand using the analytical balance. Add 19 µL of the 2000 ppm std and analyze. This value must fall within the in-house generated control limits of 86-130%.

d. Blank: Weigh out 10 mg of the dried Ottawa sand using the analytical balance and add 40 µL of the Milli-Q water and analyze. The method blank concentration should be less than the EQL. If the MB concentration is greater than the EQL, all associated samples will be re-analyzed unless the sample concentrations are greater than 20x the MB concentration. If MB concentration is between MDL and EQL, samples will be qualified with an A flag unless the sample concentration is greater than 20x the MB

concentration. If the MB fails these criteria, analysis should be stopped, and the problem should be identified and corrected before analysis can resume.

Samples

6. Weigh a well-mixed, dry, homogeneous sample into a platinum boat using an analytical balance.

NOTE: Sample size should be 10 mg.

7. Place the sample in the saddle and close the injection port.

8. Push "START" and type in the mass. Hit enter. Wait until the unit beeps before adding the sample boat into the furnace. The red light on the unit will move from WAIT to INJECT to ACQUIRE. Unit will chime when finished. Remove the boat to the solid metal block to cool before proceeding.

NOTE: Each soil sample must be analyzed with a minimum of 2 replicates. If the RPD between the 2 replicates is within 0-20%, then the average of the 2 replicates can be reported. If the RPD is >20%, a third replicate must be analyzed and the average of the three replicates would then be reported.

If Quadruplicate analysis is required for all samples. Each sample will be composed of 4 individual 10 mg analyses. Report all four individual readings and the average of the four readings. The method citation will still be SW846 9060.

For Quad analysis: The ICV, CCV, ICB, CCB, LCS and MB do not need to be read in quadruplicate, but the MS/MSD does if requested by client.

C. Shutdown
Follow SOP WCM-18.

QUALITY CONTROL

- Calibration Check Standard
Three standards are analyzed immediately after calibration to verify linearity. All must be from the same source as the calibration standard and meet the control limit of ± 10% of the true value.

- Replicates
Replicates should meet a %RPD of 20. If not, read a third aliquot of sample.

- Initial Calibration Verification (ICV)
The ICV must be run immediately after the three calibration check standards and must meet control limit of ± 10% of the true value. If not, recalibrate.

- Initial Calibration Blank (ICB)
The ICB must be analyzed after the ICV. The absolute value of the ICB must be less than the estimated quantitation limit (EQL). If the ICB is outside of the control criteria, sample analysis should not procede. Correct the problem and recalibrate the instrument.

- Method Blank (MB)
  The MB is carried through all prep procedures and analyzed with a frequency of 5%. The method blank concentration should be less than the EQL. If the MB concentration is greater than the EQL, all associated samples will be re-analyzed unless the sample concentrations are greater than 20x the MB concentration. If MB concentration is between MDL and EQL, samples will be qualified with an A flag unless the sample concentration is greater than 20x the MB concentration. If the MB fails these criteria, analysis should be stopped, and the problem should be identified and corrected before analysis can resume.

- Laboratory Control Sample (LCS)
  An LCS consisting of known concentration must be prepared and analyzed for each batch of 20 samples, and meet the current control limits of 86-130%. If the LCS recovery is outside of the control limits, the system is out of control. Terminate the analysis and identify the problem. Once the problem has been corrected, the samples should be re-prepared and analyzed.

- Continuing Calibration Verification (CCV)
  The CCV is analyzed after every 10 analytical samples It must be from a second source and must meet current control limits of ± 10% of true value. If not, the calibration procedure must be repeated and all samples since the last successful CCV must be reanalyzed.

- Continuing Calibration Blank (CCB)
  The CCB is analyzed after every CCV and must be less than the absolute value of the EQL. If not, the instrument must be re-calibrated, and all samples since the last acceptable CCB must be reanalyzed.

- Matrix Spike/Matrix Spike Duplicate
  A matrix spike/matrix spike duplicate must be performed on each group of samples of a similar matrix type with a frequency of 5%. The recovery should be within the current in-house generated control limits of 35-155%. In addition, the RPD should be less than 16. If either of the recoveries or the RPD are outside of the control limits, the results will be flagged.

Calculation:

The instrument provides calculated sample results in mg/kg; calculations are only necessary if a dilution was used.

Raw Data Value (mg/kg) x Dilution Factor = TOC (mg/kg)

ACCURACY:

A matrix spike and matrix spike duplicate must be performed on each group of samples of a similar matrix type with a frequency of 5% and meet the current control limits for accuracy.

Spike calculation:

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

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

If there is insufficient volume available for an MS/MSD, perform an LCS/LCSDUP.

PRECISION:

A matrix spike duplicate must be analyzed on each group of samples of a similar matrix type with a frequency of 5% and meet the current control limits for precision.

Use relative percent difference (RPD) calculation:

$$RPD = \frac{|MS-MSD|}{(MS+MSD)/2} \times 100$$

MS = Matrix Spike Value
MSD = Matrix Spike Duplicate Value

If there is insufficient volume available for an MS/MSD, perform an LCS/LCSDUP.

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.

SAFETY:

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

TITLE: Total Organic Carbon Analysis

DEPARTMENT: Inorganic - Wet Chemistry

APPLICATION: Determination of nonpurgeable carbon in water matrices. (TOC as NPOC).

REFERENCE: EPA 600 4-79-020, Revised March 1983, Method 415.1
EPA Manual SW-846, 3rd Edition, Method 9060
Dohrmann DC-190 High Temperature TOC Analyzer Operation Manual, Sept. 1994, Revision C

PROCEDURE SUMMARY:

The DC-190 system features a vertical quartz combustion tube packed with supported platinum catalyst which receives a continuous flow of oxygen or air at 200 cc/minute. The furnace is normally maintained at 680 °C but can be varied to any temperature up to 900 °C. Organic containing samples are manually or automatically introduced into the combustion tube via an air-actuated injection port. Through catalytic oxidation, the sample is completely oxidized to CO_2 and H_2O. The gas flow sweeps the CO_2 containing steam out of the combustion tube and into the IC reactor. It continues through a condenser, a gas liquid separator, and moisture trap. Final H_2O removal is accomplished by a permeation dryer. The dried CO_2 containing gas is then passed through a halogen scrubber and to a CO_2 specific non-dispersive infrared detector (NDIR) for peak quantification. Inorganic carbon (IC) samples are manually or automatically introduced into the IC reactor, which contains acidic water solution at room temperature, via an air-operated injection port. In this acidic environment, all forms of IC are purged out of the solution as CO_2 by the continuous flow of gas. The gas then continues through the drying system to the NDIR detector for quantitation.

LABORATORY DETECTION AND REPORTING LIMITS:

	LOD	EQL
Water:	0.33	1.0 mg/L

Approximate working range of method: LOD to 250 ppm.

REVIEWED BY: Jeffrey A. Gordon — Date: 4-19-99
Inorganic Section Supervisor

Gregory J. Graf — Date: 4-21-99
Quality Assurance Officer

APPROVED BY: Glen A. Coder — Date: 4-21-99
Laboratory Manager

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SAMPLE HANDLING & PRESERVATION:

A 125-mL sample is preserved in a glass(preferable) or plastic container at the time of collection by acidifying to pH ≤ 2 with sulfuric acid (H_2SO_4) or hydrochloric acid (HCL). The sample is then refrigerated to 4°C. Holding time for the preserved sample is 28 days from date of collection.

APPARATUS:

Dohrmann DC 190 Total Organic Carbon Analyzer
Autosampler Vials (10 mL volume)
Pipettors (0.1-100 µL, 100-1000 µL, 0.1-5.0 mL)
Syringes (50 µL, 100 - 250 µL, 1 mL)
Volumetric flasks (50 mL, 100 mL, 200 mL, 1000 mL)
Analytical balance
Top loader balance

REAGENTS:

Milli-Q deionized water
Nitric acid (HNO_3), concentrated
Potassium hydrogen phthalate (KHP), 2 sources
Oxygen gas, 99.9% purity grade
Phosphoric acid

Prepare stock standard, 2000 ppm as carbon, as follows:

- Dissolve 425 mg KHP in 100 mL Milli-Q water. Store in an amber bottle. Shelf life of one month.

Prepare calibration standard

- 5 ppm as follows: 0.25 mL of 2000 ppm to 100 mL of Milli-Q water
- 50 ppm as follows: 1.25 mL of 2000 ppm to 50 mL of Milli-Q water
- 250 ppm as follows: 6.25 mL of 2000 ppm to 50 mL of Milli-Q water
  NOTE: The Linear range is not more than +10% of highest standard
  All calibration standards listed above have a one week shelf life

PROCEDURE:

1. Start-up the instrument, specific for each matrix - soil or aqueous. (See EN CHEM SOP, WCM-18.)

2. To calibrate for TOC as NPOC, set up vials as follows:
   a. Vial #1, standard solution, no peg
   b. Vial #2, standard solution, peg on the outside
   c. Vial #3, Milli-Q water, no peg
   d. Vial #4, Milli-Q water, peg on the inside

3. Analyze 5.0, 50.0, and 250.0 mg/L standards from the same source as the calibration standard. The recovery must be ± 10%. If any recovery fails, recalibrate the instrument.

   a. Analyze an ICV sample(10 mg/L). Value must be ± 10%.
   b. Analyze an ICB sample. Value must be less than EQL.
   c. Analyze LCS sample. Value must be within current control limits .
   d. Analyze method blank (Milli-Q H_2O) sample. Value must be less than EQL.

4. Prepare samples as follows:

   a. Shake sample vigorously to suspend sediment.
   b. Pour into a clean vial for autosampler.
   c. Each sample will be composed of 3 injections. The first result in the triplicate may be omitted as this injection is used to prime the combustion tube. The average of the replicates is used for reporting.

   If referencing SW846 9060, Quadruplicate analysis is required for all samples. Each sample will be composed of 4 individual injections. Report all four individual injection readings and the average of the four readings. If the client desires to have SW846 9060 cited, but does not want Quadruplicate analyses performed, the default procedure for analysis will be followed and the method citation will be changed to SW846 9060Mod.

   For Quad analysis: The ICV, CCV, ICB, CCB, LCS and MB do not need to be read in quadruplicate, but the MS/MSD does need to be.

5. MS are prepared at 10 ppm by adding 0.025 ml of the 2000ppm stock standard to 4.975ml of sample (5.0 ml final volume).

6. The calibration established will be valid for several trays of samples up to a total of 20 samples. A new laboratory control sample (LCS) and method blank (MB), as well as additional matrix spike (MS) and matrix spike duplicate (MSD) must be added to continue samples analysis past 20.

7. Shut down instrument. (See EN CHEM SOP, WCM-18.)

QUALITY CONTROL:

- Calibration Check Standard
  Three standards are analyzed immediately after calibration to verify linearity. All must be from the same source as the calibration standard and meet the control limit of ± 10% of the true value.

- Replicates
  Replicates should meet a %RSD of 20 (unless the sample concentrations are < 5x the EQL). If not, repeat analysis, dilute sample and repeat, etc. until criteria is met.

- Initial Calibration Verification (ICV)
  The ICV must be run immediately after the three calibration check standards. It must be from a second source and meet current control limits of ± 10% of the true value. If not, recalibrate.

- Initial Calibration Blank (ICB)
  The ICB must be analyzed after the ICV and be less than the absolute difference of the estimated quantitation limit (EQL).

- Method Blank (MB)
  The MB is carried through all prep procedures and analyzed with a frequency of 5%. Rejection criteria is < LOD. Other criteria may apply, such as regulatory limit and analyte concentration in samples.

- Laboratory Control Sample (LCS)
  An LCS consisting of known concentration must be prepared and analyzed for each batch of 20 samples, and meet the current control limit.

- Continuing Calibration Verification (CCV)
  The CCV is analyzed after every 10 analytical samples and must meet current control limits of ± 10% of true value. If not, the calibration procedure must be repeated.

- Continuing Calibration Blank (CCB)
  The CCB is analyzed after every CCV and must be less than the absolute value of the EQL. If not, the calibration procedure must be repeated.

- Matrix Spike
  A spike must be performed on each group of samples of a similar matrix type with a frequency of 5%. Recovery must meet the current control limits.

- Matrix Spike Duplicate
  A matrix spike duplicate must be analyzed on each group of samples of a similar matrix type with a frequency of 5%. Recovery must meet the current control limits for accuracy and the difference between the MS and MSD must meet current control limits for precision.

CALCULATION:

The instrument provides calculated sample results in mg/L, calculations are only necessary if a dilution was used.

Raw Data Value (mg/L) x Dilution Factor = TOC as NPOC (mg/L)

ACCURACY:

A matrix spike and matrix spike duplicate must be performed on each group of samples of a similar matrix type with a frequency of 5% and meet the current control limits for accuracy. If method SW846 9060 is referenced, accuracy is to be at a frequency of 10%.

Spike calculation:

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

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

If there is insufficient volume available for an MS/MSD, perform an LCS/LCSDUP

PRECISION:

A matrix spike duplicate must be analyzed on each group of samples of a similar matrix type with a frequency of 5% and meet the current control limits for precision. If method SW846 9060 is referenced, precision is to be at a frequency of 10%.

Use relative percent difference (RPD) calculation:

$$RPD = \frac{|MS-MSD|}{(MS+MSD)/2} \times 100$$

MS = Matrix Spike Value
MSD = Matrix Spike Duplicate Value

If there is insufficient volume available for an MS/MSD, perform an LCS/LCSDUP

En Chem, Inc.

Quality Assurance Document

Standard Operating Procedure

TITLE: Volatile Organic Analysis by GC/MS - Methods 5030B/8260B

DEPARTMENT: Volatile Organics Laboratory

APPLICATION: The method outlined within is used for the detection of volatile organic compounds by GC/MS in waters and methanol extracted soils. See the accompanying Target Compound List (TCL) for the analytes to which this method applies.

REFERENCES: 40 CFR, Part 136, Appendix B, Revision 1.11

EPA SW-846, Methods 8260B, December 1996

EPA SW-846, Methods 5030B, December 1996

PROCEDURE SUMMARY:

Volatile compounds are purged out of water or medium level extracted solid matrices using a steady stream of helium. The compounds are trapped on an adsorbent trap, which is then heated rapidly, desorbing the compounds onto a GC/MS system. Separation occurs on a capillary column before entering the mass spectrometer, where spectra are generated that can be compared with previously prepared standard spectra. Quantitation is based upon analyte response verses standard response relative to internal standards.

APPROVED BY: _____ _____
Julie A. Trivedi — Date
Quality Assurance Officer

_____ 8-3-01
Al Seier — Date
Manager, Volatile Organics

_____ _____
Glen A. Coder — Date
Laboratory Manager

Annual Review

Date:						
Initials:						

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SAMPLE HANDLING AND PRESERVATION:

Water samples must be preserved with 1:1 HCl to pH <2. All samples must be stored at 4°C until analysis. Before using samples, ensure that no bubbles are found in the water samples. The sample must be flagged as having headspace if there is a bubble greater than 0.6 cm in diameter.

Samples preserved with HCl acid must be analyzed within 14 days of sampling. Samples which are not preserved must be analyzed within 7 days of sampling.

INTERFERENCES:

1) Masking:
This occurs when a compound is present at such a high concentration that its peak is broad enough to cover peaks with similar retention times.

2) Matrix Dampening:
This occurs when the sample, usually a soil, contains substances that impede the purging of volatiles out of the sample or contains high concentration of total organic carbon (TOC). This effect causes low internal standard areas and/or poor surrogate recovery.

3) Foaming:
Samples may foam during sample purging due to soaps, and surfactants. This foam will damage the sample concentrator and potentially the GC/MS. If foaming is observed during sample prep, the sample will be diluted to reduce the risk of damaging the analytical system

Initial Demonstration of Proficiency:

Each chemist must demonstrate initial proficiency with this method by generating acceptable accuracy and precision data in a clean matrix fortified with the target analytes. To validate the method the chemist must analyze four replicates of a QC check standard. The QC check standard is prepared from second source standards independent from the standards utilized to prepare the calibration curve. The average percent recovery (%R) should be within 80-120% and the relative standard deviation (%RSD) should be less than 20%.

I. APPARATUS AND MATERIALS

GC/MS- HP 5970, 5971, 5972 or 5973 MSD, or equivalent with Capillary Direct inlet. Integrated data system capable of scanning 35-300amu at 1 sec/scan.

Tekmar 3000 Concentrator

Archon 5100A Autosampler

Dynatec PTA-30 Autosampler

Capillary Gas Chromatography column - 20m x 0.18mm ID RTX-624 megabore (Restek, Inc.), with a 3µm film thickness; OR 40m X 0.18mm ID DB-624 (Restek, Inc.), with a 1.0 um film thickness.

Syringes - 5 ml Hamilton gastight with Luer-Lock tip; 5, 10, 25, 50, 100, 500, 1000 µL gastight microsyringes, 50, 100, 500ml Class A volumetric flasks.

Standard solution storage containers - 1.5mL, 7mL, 14mL amber vials with PTFE-lined screw caps; 2 ml micro reaction vials with miniert valves and replaceable septa (Supelco).

REAGENTS:

Gases: Helium, UHP grade

Methanol: Burdick & Jackson Purge-and-Trap Grade or equivalent

Organic Free Water (OFW): Organic Free Water is water with organics present below MDL for most compounds. (See IV. C.: 'Method Blank Criteria'.)

Standards: The following standards or the equivalents may be used:

Primary Standard Sources:

Restek VOC Mixes (Custom and standard stock mixes). See Table 5.
Restek Internal Standard Mix.
Restek Surrogate Standard Mix.
Restek 4-Bromofluorobenzene Solution.

Secondary Standard Sources:

Supelco Purgeable Kit Mixes.
Accu-standard Custom Kit Mixes

PROCEDURE

Chromatographic conditions

General:

Injector temperature:	220°C
Transfer line temperature:	280°C

Column:

Carrier gas (He) flow rate:	0.5 mL/min

Temperature program:

Initial temperature:	35°C and hold for 2 minutes
Ramp	8°C/min. to 180°C.
Final temperature:	Hold at 180°C for 3 min.

Purge and Trap Conditions

General:

Purge	11 minutes
Desorb	2 minutes
Bake	4 minutes

All temperature zones are set to 220°C

These are typical analytical conditions. The actual conditions used at the time of analysis may be different to allow for system optimization.

II. INITIAL CALIBRATION

Demonstration and documentation of an acceptable initial calibration is required before any samples are analyzed. Recalibration may need to be performed as indicated by results of continuing calibration check standards.

A. Verification of Instrument Performance

A 50ng BFB injection or purge must meet spectral criteria as defined in SW846 8260B or CLP OLM3.1* criteria, depending on project scope requirements (see Appendix C, Tables 1 & 2). If the tune spectrum does not meet the criteria after several injections, then the instrument may need to be retuned. The instrument must the BFB criteria before proceeding with the standard calibration.

* Note that OLM3.1 criteria is based on three scans and a background subtraction.

B. Preparation of Standards

Stock Reference Standards:

Stock standards may be prepared from neat standards and/or prepared mixes can be purchased. (See Appendix B for the Target Compound List.) A reference standard prepared from a neat standard is made up at a 10,000µg/mL concentration as follows:

Place about 9.8 ml of methanol into a 10mL ground-glass stoppered volumetric flask. Allow the flask to stand unstoppered until all alcohol-wetted surfaces have dried and weigh to the nearest 0.1mg. Using a 100µL gastight syringe, add about 20-25 drops of the neat standard to the flask to obtain about 100mg of standard. Let the drops fall just above the surface of the methanol but do not let the syringe needle touch the sides of the flask neck.

Reweigh the flask, dilute to volume, stopper, then mix by inverting the flask several times. Store without headspace in a 7mL amber vial in a freezer. All data regarding standard preparation must be entered into the standard preparation logbook. A number is assigned to each prepared standard solution and that number is entered on all logs where a solution is used. The vial containing a prepared standard solution must be labeled in accordance with the En Chem SOP, and include the assigned log number. A brief description of the standard must also appear on the label.

Working Standards:
Prepare working standards of both the volatile target compounds and surrogates at 100µg/mL in P&T methanol. Prepare an Internal Standard and an Internal Standard/Surrogate solution (ISSTD)

at a concentration of 250µg/mL in P&T methanol. Record all data regarding preparation of each standard solution in the standard logbook as described above for stock standards. Label the working standard vials as described above for the stock standards. All standards should be stored in a freezer.

Prepared standard solutions in methanol are stable for about 4 weeks when stored below -15°C except for volatile gas analytes, which are made on a weekly basis due to degradation. Standards should be replaced if it is determined that they have deteriorated.

Preparation of Calibration Standards:

An initial calibration consisting of at least five calibration points (six points are needed for quadratic regressions) is analyzed before sample analysis may proceed. The standards are prepared with all target compounds and surrogates at equal concentrations. The calibration levels are prepared with a 100ug/L working standard as follows:

Cal Level	Amt of working standards (uL)	Volumetric
1 µg/L	1 µL	100 mL
5 µg/L	5 µL	100 mL
10 µg/L	10 µL	100 mL
20 µg/L	10 µL	50 mL
50 µg/L	25 µL	50 mL
100 µg/L	50 µL	50 mL
150 µg/L	75 µL	50 mL
200 µg/L	100 µL	50 mL

The associated volumetric is inverted three times and the contents placed into a non-preserved, 40mL VOA vial, without headspace, for instrument analysis. The autosampler adds 1µL of Internal Standard mix at 250µg/mL into a 5mL aliquot, resulting in a final ISTD concentration of 50µg/L. This concentration is the same for all standards, blanks, and associated QC analyses.

A calibration curve for waters and medium level soil methanol extracts is purged at ambient temperature. Analyze these solutions using purge-and-trap and GC/MS methodology.

Preparation of Calibration File:

Identify the compounds using reference spectra and retention time data. Assign response factors to each compound using the internal standard technique based on its response in each standard. Refer to the formula sheet for the response factor formula in Appendix A, #1. See Appendix B for the assigned internal standard for each analyte.

Check linearity of response by calculating the percent relative standard deviation (RSD) of the response factors for each compound. The % RSD for each individual Calibration Check Compound (CCC) must be less than or equal to 30.0%. In addition, the minimum acceptable average response factor for System Performance Check Compounds (SPCC) must meet specified Response Factor (RF) criteria. (See Appendix C, Table 3 for lists of CCC and SPCC compounds.)

The validity of the calibration is accepted or rejected after making these comparisons. Some common causes of a bad calibration curve are: standard degradation, poor instrument stability or improper purge flow rate.

Any target analyte with an RSD of less than 15% is considered valid and the average response factor may be used for quantitation purposes. If the %RSD exceeds 15%, the analyst must choose the best calibration option for quantitation purposes. Linear, and Quadratic regression and third order polynomial are the other options used for analyte quantitation.

In order to consider the initial calibration acceptable, an Initial Calibration Verification Standard (ICV) must be analyzed before samples analysis, preferably 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 (CCV) standard before the initial calibration may be considered valid.

If the calibration is not acceptable, the appropriate calibration solution(s) is reanalyzed to obtain acceptable % RSD and RFs over the entire calibration range.

III. DAILY CHECKS AND CONTINUING CALIBRATION CHECK

A BFB tune, continuing calibration check standard, method blank, and LCS must be performed at the beginning of each 12 hour period during which sample analyses are performed.

A. Daily BFB Tune

To check the tune, follow the procedure in section II A : Verification of Instrument Performance. If the tune has failed, consider modifying the tune file again as described in section II. A, above. If the BFB or the autotune fails repeatedly, source cleaning may be necessary. The BFB tune must meet the spectrum criteria as defined in Appendix C, Table 1, before proceeding with the continuing calibration check standard.

B. Continuing Calibration Check Standard

Prepare a 50µg/L standard by adding 25µL of the target compound working standard at 100µg/mL into a 50mL volumetric of organic free water. The autosampler will then add 1µL of ISSTD mix at 250µg/mL into a 5mL aliquot. Compare the daily response factors with the average response factors from the initial calibration. In addition, the Relative Retention Time for each analyte must compare within ± 0.06 RRT units of the RRT established in the initial calibration.

The percent difference for any CCC compound may not be greater than 20.0%. Some target analytes, such as the Ketones, have extremely poor purging efficiencies and may have a higher relative percent difference. If any non-CCC compounds have a percent difference greater than 20.0%, analyst discretion is used before sample analysis occurs.

The daily response factor of any SPCC compound must be within the criteria (as listed in Appendix C, Table 3). Check the validity of the ID file's qualitative matches. If the SPCC or CCC conditions are not met, the standard is not valid and should be repeated. Repeated failure to meet acceptance criteria is a sign that the initial calibration curve is no longer valid. If no extraneous causes of standard failure are suspected, the initial calibration must be repeated. Refer to II.B: 'Preparation of Standards', above.

C. Method Blank

A method blank consists of organic free water. The method blank is analyzed in the exact manner that the samples are analyzed. In addition to the internal standard and surrogate QA/QC requirements of sample analysis, the method blank must meet the following criteria before sample analysis may begin:

1. No common solvent (Acetone, Acetonitrile, 2-Butanone, 2-Hexanone, 4-Methyl-2-pentanone, Methylene chloride, and Tetrahydrofuran) may be present above 5 times the EQL (Estimated Quantitation Limit).

2. Target compounds other than common solvents should not be greater than the reporting limit.

3. Non-target compound peaks with areas greater than 10% of the area of the nearest internal standard, other than surrogates, must not be present in the chromatogram if tentatively identified compounds (TICS) are requested.

If contaminants are found in the blank, an attempt should be made to identify and minimize/eliminate the source.

D. Lab Control Sample

A lab control sample (LCS) and lab control duplicate (LCSD) are performed within every 12 hr. analytical clock to check for instrument accuracy and precision. Prepare a 50µg/L LCS by adding 25µL of the second source spike calibration mix at 100µg/mL into a 50mL volumetric. Invert the volumetric three times slowly and pour contents into a 40mL non-preserved VOA vial. Place on the autosampler. The autosampler then adds 1uL of ISSTD mix @ 250µg/mL into a 5mL aliquot.

NOTE: All limits are taken from CLP OLM3.0 criteria. An LCS for all compounds may be required based on project requirements and scope. If an LCS with all target analytes is requested, an LCSD may not be analyzed. The calibration mix used for the full spike LCS is the same used for the MS/MSD.

E. Blank Spike/Blank Spike Duplicate

A blank spike (BS) /blank spike duplicate (BSD) will be used for QC control in methanol extracted soils if Matrix Spike/Matrix Spike Duplicates are not analyzed because of insufficient sample amounts. A BS/BSD is made by weighing 4g of Ottawa sand and 4mLs of P & T methanol into a 20mL scinillation vial. 4uL of the surrogate mix @ 2500µg/mL and 125µL of a second source spike mix @ 100µg/mL is spiked into the scintillation vial giving the surrogates and spikes a final concentration of 50µg/L.

A 1mL methanol aliquot of the sample is placed into a 50mL volumetric and brought to volume with OFW. The volumetric is inverted three times slowly, poured into a non-preserved 40mL VOA vial and placed on the instrument for analysis. The autosampler then adds 1µL of the ISTD mix @ 250µg/mL into a 5mL sample aliquot for a final ISTD concentration of 50µg/L.

Due to poor purging efficiencies of some compounds found in the spike mixes, a default percent recovery criteria of 70 to 130 has been established.

IV. SAMPLE ANALYSIS

A. Identify Matrix

The two matrices are water and methanol extracted soils. A methanol extract is required if a 5.0 g sample size of soil yielded results above the calibration range.

B. Sample Preparation

Samples must be warmed to ambient temperature before analysis begins.

WATER:

1. Place a 40mL VOA vial of the specified sample onto the autosampler. Transfer a 5mL aliquot of the sample into a sparge tube and purge.

Primary Dilution (1:10 or less) - 500µL to 2.5mL of sample is added into OFW on the autosampler for a final volume of 5mL.

3. Secondary Dilution (1:20 to 1:10,000) - 5.0µL to 2.5mL of sample is added into a 50mL volumetric brought to volume with OFW. Invert slowly three times. Pour the contents into a non-preserved 40mL VOA vial and place on the instrument for analysis.

To each 5mL sample aliquot, the autosampler adds 1µL of the ISSTD mix at 250µg/L, resulting in a final concentration of 50µg/L.

SOIL:

Medium Level Extracts

A 25g En Core sampler is extruded into 20mLs of Purge and Trap Methanol in a 60mL soil jar. The tare weight of the jar and the approximate weight of 20mLs of MEOH (15.8g) is added and recorded. The jar containing the 25g plug then placed on the balance and the tare weight recorded. The final sample weight is found by subtracting the weight of the jar/MEOH from the weight of the jar/MEOH/sample. Methanol is then added to bring the final ratio of soil to MEOH to 1:1. The amount of surrogate @ 2500µg/mL added is equal to the final weight of the sample/MEOH.

A 5g En Core may also be used in place of a 25g En Core sampler. It will follow the same procedure as mentioned above except the sample will be extruded into a 20mL scinillation vial containing 3mLs of P&T MEOH. The most important aspect is to maintain a 1:1 ratio of soil to methanol.

Samples received pre-weighed in the field should also have a 5g or 25g sample aliquot already preserved in MEOH. The same procedure for medium level preparation, as listed above, is followed.

QC samples are prepped per 20 samples. A MEOH blank is made by weighing 5g of Ottawa sand into a 20mL scintillation vial and adding 5mLs of P & T methanol. In order to obtain a final concentration of 50µg/L for surrogates, 5µL of surrogate mix @ 2500µg/mL is spiked into the scintillation vial. The same process is used for Blank Spikes/Blank Spike Duplicates* except 4g of Ottawa sand and 4mLs of P & T methanol are placed into a 20mL scintillation vial. 4µL of the surrogate mix @ 2500µg/mL and 125µL of a second source spike mix @ 100µg/mL is spiked into the scintillation vial giving the surrogates and spikes a final concentration of 50µg/L.

Place a 1mL methanol aliquot of the sample into a 50mL volumetric and bring to volume with OFW. Slowly invert the volumetric three times, pour into a non-preserved 40mL VOA vial, and place on the instrument for analysis. The autosampler then adds 1µL of the ISTD mix @ 250µg/mL into a 5mL sample aliquot for a final ISTD concentration of 50µg/L.

Record all data pertinent to medium level soil extract preparation in the designated logbook.

* The BS/BSD is used when MS/MSD samples are not available due to insufficient sample amounts.

C. Analysis

Analyze samples by purge-and-trap GC/MS methodology. See En Chem SOP for proper instrument operating conditions. Waters and medium level soils are purged at ambient temperature.

Identify and quantitate the target compounds using relative retention times (RRT) and average response factors from the 5-point calibration curve. For establishing correspondence of the RRT, the sample component RRT must compare within +/- 0.06 RRT units of the RRT of the continuing calibration standard component. Compare computer-matched compounds with reference spectra to accept or reject each identification. All ions present in the reference spectrum that are at least 10% of the base peak must be present in the sample background-subtracted spectrum. Also, the relative intensities of these ions must agree within +/- 20% between the standard and sample spectra. While this is a good guideline, acceptance or rejection will depend upon the judgment of the analyst.

It may be necessary to adjust the volume of sample used for analysis so that the concentration of the analytes of interest are within the calibration range. A maximum of 5.0mL of an aqueous sample will be analyzed. While every attempt is made to provide the best detection limits, the most concentrated analysis of a sample is often limited by interfering peaks of non-target compounds. Detection limits will be raised to account for the dilution.

D. Verification of Sample Preservation

Following sample analyses, the pH of all waters shall be verified by wide range pH paper. The results will be documented in the logbook. If the pH is found to be 3 or greater, and the sample was not analyzed within 7 days of collection, a narrative is written and submitted with the sample results.

Quality Control:

The surrogate recoveries and internal standard areas must be checked to determine that they are within the established control limits. If the requirements are not met, the sample is reanalyzed. The internal standard areas for the method blank and samples analyzed must be within -50% to +100% of the calibration check standard's internal standard areas. In addition, the retention time must be within ± 30 seconds from the last calibration check. If an internal standard is outside of control limits but no compounds of interest are being quantified, no re-analysis is necessary. If the second analysis is within control limits, the data from that analysis is reported. If the failure is repeated, data from the "better" of the two analyses is reported and the anomaly is discussed in a sample narrative.

A sample matrix spike (MS) and matrix spike duplicate (MSD) must be performed every 14 days or 20 samples per instrument of the same matrix type by the same method and target compound list. A matrix type for an MS/MSD is defined as an unheated water which includes medium level soil methanol extracts. The spiking solution must contain all constituents of concern and be from a secondary source other than the reference standards used in the calibration. The percent recoveries

for each spiked compound must be checked to determine that they are within the established control limits. If these are not met, the sample MS and/or MSD is reanalyzed.

E. Annual Quality Control

1. Method Detection Limits (MDLs):
MDLs are determined as specified in 40 CFR, Part 136, Appendix B, and must be performed once a year per instrument for all target compounds of interest. Each available instrument will perform one unheated set of MDLs using Purge and Trap methodology. At least seven replicates will be analyzed per set.

2. Surrogate Recovery Limits:

One set each of surrogate recovery limits for unheated waters and methanol extracted soils must be established for all instruments performing GC/MS analysis. These limits are to be updated annually using data from all GC/MS instruments.

3. Matrix Spike and Duplicate (MS/MSD) Recovery Limits:

One set each of MS/MSD recovery limits for unheated waters and methanol extracted soils must be established for all instruments performing GC/MS analysis. These limits are to be updated annually using data from all GC/MS instruments.

METHOD EXCEEDANCES

Allowance is made for sporadic exceedances of control limits in LCS/LCSD and MS/MSD without further corrective action. Data for the analytes that exceed the control limits shall be qualified. The number of allowances is based on the number of analytes spiked. More information can be found in Appendix I of this SOP.

DETERMINATION OF SPORADIC MARGINAL FAILURES ALLOWED

N[1]	X
5 - 15	1
16 - 30	2
31 - 45	3
46 - 60	4
61 - 75	5
76 - 90	6
91 - 105	7

N = Number of target analytes spiked.
X = Number of Sporadic Marginal Failures (SMF) allowed

[1] = The number of SMF allowances depend upon the number of target analytes reported from the analysis. For instance, if the full list of target componds as presented in Appendix A are reported, then five (5) SMFs are allowed. If the Matrix Spike (MS) and/or the Laboratory Control Spike (LCS) includes only a subset of compounds and for surrogates, allow up to one (1) SMF for each B/N and A grouping. B = Base, N = Neutral and A = Acid compounds.

NOTE: SMF's are used when QC limits have been established. They are not used for compounds with advisory QC limits (i.e., QC limits have not yet been established).

APPENDIX A

FORMULA SHEET

#1. Response Factor:

$$RF = \text{RESPONSE FACTOR} = \frac{(Ax)}{(Ais)} \times \frac{(Cis)}{(Cx)}$$

Where:

Ax = Area of analyte's characteristic ion of quantitation

Ais = Area of corresponding internal standard's ion of quantitation

Cis = Concentration of corresponding internal standard

Cx = Concentration of analyte to be measured

#2. Concentration calculation for raw amount using average RF and internal standards:

$$\text{Sample Amount} = \frac{(Ax)}{(Ais)} \times \frac{(Cis)}{(\text{Average RF})} \times DF$$

Where:

Ax = Area of analyte's characteristic ion of quantitation

Ais = Area of corresponding internal standard's ion of quantitation

Cis = Concentration of corresponding internal standard

Average RF = Average Response Factor of compound from initial calibration

DF = Dilution Factor (if applicable)

APPENDIX B

TARGET COMPOUND LIST, INTERNAL STANDARDS, SURROGATES, AND ESTIMATED QUANTITATION LIMITS (EQLs) *

	LOW LEVEL EQL (ug/l)	MEDIUM LEVEL EQL (ug/Kg)
INTERNAL STANDARD PENTAFLUOROBENZENE:		
Dichlorodifluoromethane	2	50
Chloromethane	2	50
Bromomethane	2	50
Vinyl chloride	2	50
Chloroethane	2	50
Trichlorofluoromethane	2	50
Dichlorofluoromethane	2	50
Allyl Chloride	5	250
Acrolein	10	500
Acetone	5	150
Diethyl Ether	1	50
1,1-Dichloroethene	1	50
Iodomethane	5	250
Acrylonitrile	10	500
Methylene chloride	1	50
Carbon disulfide	1	50
Trans-1,2-Dichloroethene	1	50
Methyl Tert Butyl Ether	1	50
1,1-Dichloroethane	1	50
Vinyl Acetate	10	500
2-Butanone	5	150
Diisopropyl Ether	1	50
Cis-1,2-Dichloroethene	1	50
Bromochloromethane	1	50
Chloroform	1	50
2,2-Dichloropropane	1	50
Tetrahydrofuran	10	500
1,2-Dichloroethane	1	50
1,1,1-Trichloroethane	1	50
1,1-Dichloropropene	1	50
Carbon tetrachloride	1	50
Benzene	1	50
Diethoxymethane	10	500
Dibromofluoromethane (SURR)		

APPENDIX B (Continued)

TARGET COMPOUND LIST, INTERNAL STANDARDS, SURROGATES, AND ESTIMATED QUANTITATION LIMITS (EQLs)

	LOW LEVEL EQL (ug/l)	MEDIUM LEVEL EQL (ug/Kg)
INTERNAL STANDARD 1,4-DIFLUOROBENZENE:		
Trichloroethene	1	50
1,2-Dichloropropane	1	50
2,3-Dichloropropane	1	50
Dibromomethane	1	50
Bromodichloromethane	1	50
2-chloroethyl vinyl ether	5	250
cis-1,3-Dichloropropene	1	50
Toluene	1	50
trans-1,3-Dichloropropene	1	50
4-Methyl-2-pentanone	5	150
1,1,2-Trichloroethane	1	50
Tetrachloroethene	1	50
1,3-Dichloropropane	1	50
Dibromochloromethane	1	50
2-Hexanone	5	150
1,2-Dibromoethane	1	25
Toluene-d8 (SURR)		
INTERNAL STANDARD CHLOROBENZENE-d5:		
Chlorobenzene	1	50
1,1,1,2-Tetrachloroethane	1	50
Ethylbenzene	1	50
m-, p-, Xylene	2	100
o-Xylene	1	50
Styrene	1	50
Bromoform	1	50
Isopropylbenzene	1	50
Trans-1,4-Dichloro-2-Butene	5	250
Cis-1,4-Dichloro-2-Butene	5	250
Bromobenzene	1	50
4-Bromofluorobenzene (SURR)		

APPENDIX B (Continued)

TARGET COMPOUND LIST, INTERNAL STANDARDS, SURROGATES, AND ESTIMATED QUANTITATION LIMITS (EQLs)

	LOW LEVEL EQL (ug/l)	MEDIUM LEVEL EQL (ug/Kg)
INTERNAL STANDARD **1,4-Dichlorobenzene:**		
1,1,2,2-Tetrachloroethane	1	50
1,2,3-Trichloropropane	1	50
n-Propylbenzene	1	50
2-Chlorotoluene	1	50
4-Chlorotoluene	1	50
1,3,5-Trimethylbenzene	1	50
Tert-Butylbenzene	1	50
1,2,4-Trimethylbenzene	1	50
Sec-Butlybenzene	1	50
1,3-Dichlorobenzene	1	50
1,4-Dichlorobenzene	1	50
1,2,3-Trimethylbenzene	1	50
P-Isopropyltoluene	1	50
1,2-Dichlorobenzene	1	50
N-Butylbenzene	1	50
1,2-Dibromo-3-chloropropane	5	250
1,2,4-Trichlorobenzene	1	50
Naphthalene	1	50
Hexachlorobutadiene	1	50
1,2,3-Trichlorobenzene	1	50

*Lower quantitation limits may be obtained depending on project requirements.

APPENDIX C

TABLE 1
GC/MS PERFORMANCE STANDARD

Bromofluorobenzene (BFB)

m/z	Ion Abundance Criteria
50	15-40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5-9% of mass 174
176	95-101% of mass 174
177	5-9% of mass 176

TABLE 2
GC/MS PERFORMANCE STANDARD
CLP OLM3.1
Bromofluorobenzene (BFB)

m/z	Ion Abundance Criteria
50	8-40% of mass 95
75	30-66% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	Less than 2% of mass 174
174	50- 120% of mass 95
175	4-9% of mass 174
176	93-101% of mass 174
177	5-9% of mass 176

TABLE 3

Calibration Check Compounds (CCC)

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

The % RSD for the initial calibration must be less than 30%.
The % RSD for the continuing calibration must be less than 20%.

System Performance Check Compounds (SPCC)	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
1,1,2,2-Tetrachloroethane	0.30
Chlorobenzene	0.30

TABLE 4
Chromatographic Retention Times and Characteristic Masses (m/z)

Compound	Retention Time	Primary Ion	Secondary Ions
Dichlorodifluoromethane	2.33	85	87
Chloromethane	2.54	50	52
Vinyl Chloride	2.64	62	64
Bromomethane	2.97	94	96
Chloroethane	3.05	64	66
Trichlorofluoromethane	3.23	151	101,153
Acrolein	3.64	56	55,58
Iodomethane	3.85	142	127,141
Acetonitrile	4.05	41	40,39
Carbon Disulfide	3.90	76	78
Methyl-t-butyl ether	4.21	73	57
Allyl Chloride	3.97	76	41,39,78
Methylene Chloride	4.09	84	86,49
1,1-Dichloroethene	3.68	96	61,63
Diethyl Ether	3.45	74	45,59
Acetone	3.75	58	43
trans-1,2-Dichloroethene	4.26	96	61,98
Acrylonitrile	4.31	53	52,51
1,1-Dichloroethane	4.61	63	65,83
Vinyl Acetate	4.58	43	86
2,2-Dichloropropane	5.04	77	97
2-Butanone	5.05	72	43
cis-1,2-Dichloroethene	5.05	96	61,98
Propionitrile (ethyl cyanide)	5.19	54	52,55,40
Chloroform	5.27	83	85

Compound	Retention Time	Primary Ion	Secondary Ions
Bromochloromethane	5.26	128	49,130
Methacrylonitrile	5.28	41	67,39,52,66
1,1,1-Trichloroethane	5.42	97	99,61
Carbon Tetrachloride	5.53	117	119
1,1-Dichloropropene	5.54	75	110,77
Benzene	5.71	78	-
1,2-Dichloroethane	5.77	62	98
Trichloroethene	6.21	95	97,130,132
1,2-Dichloropropane	6.44	63	112
Bromodichloromethane	6.64	83	85,127
Dibromomethane	6.55	93	95,174
Methyl Methacrylate	6.42	69	41,100,39
1,4-Dioxane	6.54	88	58,43,57
2-Chloroethylvinyl ether	6.85	63	65,106
4-Methyl-2-pentanone	7.10	100	43,58,85
Toluene	7.27	92	91
trans-1,3-Dichloropropene	7.49	75	77,39
cis-1,3-Dichloropropene	7.01	75	77,39
1,1,2-Trichloroethane	7.67	83	97,85
Ethyl Methacrylate	7.46	69	41,99,86,114
2-Hexanone	7.83	43	58,57,100
Tetrachloroethene	7.74	164	129,131,166
1,3-Dichloropropane	7.84	76	78
Dibromochloromethane	8.03	129	127
1,2-Dibromoethane	8.17	107	109,188
Chlorobenzene	8.56	112	77,114
1,1,1,2-Tetrachloroethane	8.64	131	133,119
Ethylbenzene	8.61	91	106

Compound	Retention Time	Primary Ion	Secondary Ions
m/p-Xylene	8.72	106	91
o-Xylene	9.11	106	91
Styrene	9.14	104	78
Bromoform	9.38	173	175,254
Isopropylbenzene(Cumene)	9.44	105	120
cis-1,4-Dichloro-2-butene	9.58	75	53,77,124,89
1,1,2,2-Tetrachloroethane	9.80	83	131,85
Bromobenzene	9.83	156	77,158
1,2,3-Trichloropropane	9.87	75	77
n-Propylbenzene	9.84	91	120
2-Chlorotoluene	9.99	91	126
trans-1,4-Dichloro-2-butene	9.86	53	88,75
1,3,5-Trimethylbenzene	10.00	105	120
4-Chlorotoluene	10.10	91	126
Pentachloroethane	10.47	167	130,132,165,169
1,2,4-Trimethylbenzene	10.39	105	120
sec-Butylbenzene	10.55	105	134
tert-Butylbenzene	10.32	119	91,134
p-Isopropyltoluene	10.68	119	134,91
1,3-Dichlorobenzene	10.74	146	111,148
1,4-Dichlorobenzene	10.83	146	111,148
n-Butylbenzene	11.09	91	92,134
1,2-Dichlorobenzene	11.23	146	111,148
1,2-Dibromo-3-chloropropane	12.04	75	155,157
1,2,4-Trichlorobenzene	12.84	180	182,145
Hexachlorobutadiene	12.93	225	223,227
Hexachloroethane	11.44	201	166,199,203
Naphthalene	13.12	128	-

Reagent	Conc.	Purity	Manufacturer	Vendor
502.2 Cal 2000 Megamix	2000 ug/mL	99%	Restek	Restek
2-Chloroethylvinyl Ether	2000 ug/mL	99%	Restek	Restek
502.2 Calibration Mix#1	2000 ug/mL	99%	Restek	Restek
Custom Cyclohexanes/Methyl Acetate Mix	10000 ug/mL	99%	Restek	Restek
1,4-Dioxane Standard	2000 ug/mL	99%	Restek	Restek
Diethoxymethane	neat	99%	Aldrich Chemical	Aldrich Chemical
Vinyl Acetate	neat	99+%	Aldrich Chemical	Aldrich Chemical

TABLE 6

Standard	Acronym	Concentration of Intermediate	Reagents Used	Final Volume
4-Bromofluorobenzene	BFB	25 ug/mL	50µL of 5000 ug/mL BFB into methanol	10 mL
Internal Standard	IS	250 ug/mL	5000 µL of 2500 ug/mL IS into methanol	50 mL
Surrogate Standard	SS	250 ug/mL	5000 µL of 2500 ug/mL SS into methanol	50 mL
Internal/Surrogate Stnd.	IS/SS	250 ug/mL	5000 µL of 2500 ug/mL IS/SS into MeOH	50 mL
100 ug/mL Calibration Stnd.	CAL	100 ug/mL	1250 µL of 2000 ug/mL Custom Voa Mix 1250 µL of 2000 ug/mL 502.2 Cal 2000 1250 µL of 2000 ug/mL 2-Chloroethylvinyl ether 1250 µL of 2000 ug/mL 502.2 Cal. Mix #1 125 µL of 20000 ug/mL Custom Acrolein 500 µL of 5000 ug/mL VOA Cal. Mix #1 263 µL of 9520 ug/mL Vinyl Acetate 246 µL of 10168 ug/mL Diethoxymethane	25 mL
Vinyl Acetate	VA	9520 ug/mL	0.238 mg of neat Vinyl Acetate	25 mL

INSTRUMENT OPERATING PROCEDURE

INSTRUMENT TYPE:	Apollo 9000 Total Organic Carbon (TOC) Analyzer
MANUFACTURER:	Tekmar Dohrmann
MODEL:	Apollo 9000
SERIAL NO.:	Apollo 9000 Electronics Module 99174002
DEPARTMENT:	Inorganics - Wet Chemistry

PROCEDURE:

The Apollo 9000 TOC Analyzer may be set up in different ways to accommodate the analysis of waters or soils. Analysis for these two matrices will be dealt with in separate sections.

I. Waters TOC configuration

A. Routine Operation

1. The unit will be in standby mode prior to start up. The unit and detector remain "on" at all times.
2. Check levels of acid in vials at position 70 and 1011. If either are under half full fill with 21% phosphoric acid reagent.
3. Check level of Milli-Q H_2O in 1L amber bottle. If under half full refill with fresh Milli-Q H_2O.
4. Check the tubing to make sure that the Cu In tubing is connected to the copper side of the U-shaped scrubber.
5. Turn on O_2 valves in this order ONLY: Open the tank valve, then SLOWLY open the regulator valve. Doing the reverse may blow out the valves.
6. Log on to computer which interfaces with the Apollo 9000. Click on icon named "Apollo 9000". Machine will initialize at this point.
7. Click on the Setup drop down menu and select Instrument.
8. In the Instrument Setup/Status screen enter user name, click "Ready", verify gas flow to furnace (should be 200 cc/min. +/- 20%, it can be adjusted at the regulator), make sure that "Autosampler Sparge" option is not checked and choose "with Autosampler" for Sample Introduction Preferences.
9. Before beginning any analysis verify that the sparge tube and water trap are empty, if not empty via menu commands under Setup, Diagnostics, Syringe.
10. Select calibration curve by clicking menu command Setup, Calibration, Set Active, TOC Curve. Choose the most current or new TOCW# calibration curve for the "1-20, 1-400" range.
11. Click menu command Run.

12. Click on "Sample Setup" button, then click menu command File, Open. Open the "blank.set" file. Verify the Method ID for the application you will be using and change status to "ready" for the two samples and click Save/Use.
13. Load a vial of Milli-Q H_2O in tray position #1. Click "Start". Blank run will take approximately 1 hour. When complete, record the three readings for the TOC Blank and enter them under menu option Results, Sample Blanks.
14. The unit is now ready to start analysis for aqueous TOC samples.

B. Calibration - If calibrating use the following steps, if calibration is good from previous day (verified by a check standard) move on to section C.

1. Click menu command Setup, Calibration, Standards. Open old calibration set, save standards as TOC# using the next available number. Click "OK".
2. Click menu command Setup, Calibration, Set Active, desired curve (TOC curve for TOC calibration, TC Curve for TC calibration).
3. Click on menu option Run. Click "Sample Setup" button. Open previous calibration file. Alter Sample Type, Method ID, and Reps to reflect analysis being performed. Reset Status to Ready. Save file as TOCWCAL# using the number used for calibration standards. Click "Save/Use" button.
4. Load tray with vials filled with calibration standards. Since calibration will be sparged internally, vials may be filled to the top. Click "Start" button.
5. Calibration takes approximately 3 hours. When complete the Calibration Curve screen will automatically open. Check the use box next to each of the standards and click the Recalc button. This will create a seven point calibration curve. Curve will be 1st order linear. The r-sqr value must be greater than 0.995, if not, recalibrate.

C. Sample Analysis

1. Click menu command Setup, Instrument. Check "Autosampler Sparge Option".
2. Click menu command Run. Then click "Sample Setup" button. The old schedule that appears may be edited, or a new schedule may be started. Fill in information for Sample ID, Method ID, Reps, and Status. Sample type should always be "Sample". Be certain that Method ID and Reps reflect the test being performed. Status should be "Ready" for all samples.
3. Save the schedule using the format YYMMDDW. Click Save/Use.
4. Load the tray corresponding to the schedule. Vials should be 2/3-3/4 full. The space is necessary since sparging will occur inside the vial.
5. Click Start. Sample analysis takes approximately 1/2 hour per sample.

D. Shutdown

If on the final run for the day, click menu command Setup, Instrument and make sure that the options for "Auto Shutdown" is checked.

Close Apollo 9000 software. Log off computer if no further applications are to be performed.

Turn the carrier gas off. Turn the regulator valve off, although the tank may be left on.

Empty vials. Check and dump waste collection carboy if necessary.

DO NOT turn off the unit. It will remain in a standby setting.

II. Soils TOC (STOC) configuration

A. Routine Operation - Beginning with unit in Shutdown mode

1. Open the door and take the "Cu In" tube out of the copper side of the tin/copper scrubber. Note its position prior to removing (i.e. how much tube is actually inside the U tube). Connect this tube to the black shrink wrap on the teflon tube coming from the boat sampler.
2. Place the tube and septa into the sparger on the soils unit. NOTE: Remove after analyzing soils - H_2O will travel back into the furnace if this is not done
3. Turn boat gas and sparge gas on (remember to turn off when done)
4. Turn power on (black switch). Indicator light shows when the boat sampler furnace is warmed up.
5. Turn on O_2 valves in this order ONLY: Open the tank valve, then SLOWLY open the regulator valve. Doing the reverse may blow out the valves.
6. Log on to computer which interfaces with the Apollo 9000. Click on icon named "Apollo 9000". Machine will initialize at this point.
7. Click on the Setup drop down menu and select Instrument.
8. In the Instrument Setup/Status screen enter user name, click "Ready", verify gas flow to furnace (should be 200 cc/min. +- 20%, it can be adjusted at the regulator), and choose "without Autosampler" for Sample Introduction Preferences. Make sure that the box for "Auto Shutdown" is not checked.
9. Select calibration curve by clicking menu command Setup, Calibration, Set Active, TOC Curve. Choose "Boat Calibration" for ranges 0-20, 1-400 and 100-4000. Click OK.
10. Click menu command Run.
11. Samples should be prepared as described in the SOP for soils. A homogeneous mix should be achieved. Have the sample or the standard ready to inject: that is, weighed or aliquoted, in the boat, the boat in the carrier with the lid closed, prior to clicking the "Start" button.

B. Calibration

1. Click menu command Setup, Calibration, Standards. Then click menu command File, Open and select the boat calibration curve. Click "OK".
2. Click Run, Sample Setup. Change sample type to TOC Standard. Select 1000-4000 range. Highlight Milli-Q H_2O and click exit. Set number of repeats to 1 and verify that the Method ID is Boat Sampler. Click Save/Use.
3. Click Start. Enter sample weight and follow the prompts to inject the boat.
4. Repeat steps 2 and 3 for the 1000, 2000 and 3500 standards, highlight the appropriate Standard ID for each calibration point.

5. When four standards have been read click on menu command Results, Calibration. Select the default calibration curve ID. Locate and check the four readings just performed, all others should not be checked. Click "Recalc" button. This will create a four point calibration curve. The curve will be 1st order linear. The r-sqr value must be greater than 0.995, if not, recalibrate.

C. Sample Analysis

1. Click menu command Run. Click "Sample Setup" button. Enter the sample ID, Sample type should read "sample". Choose the number of repeats for the sample and verify that the Method ID is Boat Sampler. Click "Save/Use" button.
2. Click "Start" button. Enter the sample weight. Follow the prompts to inject the boat.
3. Repeat steps 1 and 2 for each sample being analyzed.

D. Shutdown

1. Remove boat from slide.
2. Turn the boat gas and sparge gas off. Turn the carrier gas off if no further analysis of waters is to be performed. Turn the regulator valve off though the tank may be left on.
3. Remove the tube from the gas sparger tube on the soils unit.
4. Turn the power off on the soils unit (black switch)
5. Open the door of the Apollo 9000 and remove the "Cu In" tube from the outer black shrink tubing. Place this tube into the copper side of the tin/copper scrubber. Return the tube in the copper scrubber to its position prior to removal. This is important for gas circulation in the tube.
6. The fan of the soils unit will always be on in order to cool the furnace.
7. Click menu command Setup, Instrument. Click on the "Sleep" button.
8. Close Apollo 9000 software. Log off computer if no further applications are to be performed.

III. Routine/Preventative Maintenance

Refer to the manual for troubleshooting and any routine maintenance.

APPROVED BY: _____ 8/3/01
Michael J Helmann — Date
Wet Chemistry Group Leader

_____ _____
Julie A Trivedi — Date
Quality Assurance Officer

_____ _____
Glen A. Coder — Date
Laboratory Manager

Annual Review

Date:					
Initials:					

STANDARD OPERATING PROCEDURE

TITLE: Total Organic Carbon Analysis, Apollo 9000

DEPARTMENT: Inorganic - Wet Chemistry

APPLICATION: Determination of nonpurgeable carbon in water matrices (TOC as NPOC).

REFERENCE: EPA 600 4-79-020, Revised March 1983, Method 415.1
EPA Manual SW-846, 3rd Edition, Method 9060
Apollo 9000 TOC Combustion Analyzer User Manual, 1999-2000.

PROCEDURE SUMMARY:

Carbon in the sample is first converted to CO_2 by the combustion furnace for TOC and TC analysis or by the IC sparger for IC analysis. A carrier gas then sweeps the derived CO_2 through a nondispersive infrared (NDIR) detector. Sensitive to the absorption frequency of CO_2, the NDIR generates a non-linear signal that is proportional to the instantaneous concentration of CO_2 in the carrier gas. That signal is then linearized and integrated over the sample analysis time. The resulting area is then compared to stored calibration data and sample concentration in parts-per-million (ppm) is calculated.

APPROVED BY: _____ 8/3/01
Michael J Helmann — Date
Wet Chemistry Group Leader

_____ _____
Julie A Trivedi — Date
Quality Assurance Officer

_____ _____
Glen A. Coder — Date
Laboratory Manager

Annual Review

Date:					
Initials:					

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SAMPLE HANDLING & PRESERVATION:

A 125-mL sample is preserved in a glass or plastic container at the time of collection by acidifying to pH ≤ 2 with sulfuric acid (H_2SO_4) or hydrochloric acid (HCl). The sample is then refrigerated to 4°C. Holding time for the preserved sample is 28 days from date of collection.

APPARATUS:

Apollo 9000 Total Organic Carbon Analyzer
40 mL VOA vials
Pipettors (0.1 mL, 0.5 mL, 0.1-5.0 mL)
Volumetric flasks (100 mL, 200 mL, 500 mL)
Analytical balance capable of 0.1 mg resolution and accuracy

REAGENTS:

Milli-Q deionized water
Potassium hydrogen phthalate (KHP), 2 sources
Oxygen gas, 99.9% purity grade
Phosphoric acid

Prepare stock standard, 2000 ppm as carbon, as follows:

Dissolve 950 mg in 200 mL of Milli-Q water. Store in an amber bottle. Shelf life of one month.

Prepare Calibration standards as follows:

1 ppm as follows: 0.05 mL of stock standard dilute to 100 mL with Milli-Q water
10 ppm as follows: 0.5 mL of stock standard dilute to 100 mL with Milli-Q water
50 ppm as follows: 2.5 mL of stock standard dilute to 100 mL with Milli-Q water
150 ppm as follows: 7.5 mL of stock standard dilute to 100 mL with Milli-Q water
300 ppm as follows: 15.0 mL of stock standard dilute to 100 mL with Milli-Q water
400 ppm as follows: 20.0 mL of stock standard dilute to 100 mL with Milli-Q water

Prepare the ICV/CCV solution at 50 ppm as follows:
2.5 mL of stock standard from a 2nd source diluted to 100 mL with Milli-Q water.
NOTE: The Linear Range is not more than +10% of highest standard.
All calibration standards listed above have a one week shelf life.

Prepare 21% Phosphoric Acid as follows:
Add 50 mL of 85% Phosphoric acid to 100 mL DI water. Dilute to 200 mL. Shelf life = 1 year.

PROCEDURE:

1 Start-up instrument and calibrate (if necessary), as explained EN CHEM SOP, WCM-69.

2 Prepare samples as follows:
a) Shake sample vigorously to suspend sediment.
b) Pour into a clean 40 mL vial and place in autosampler.
c) Each sample will be composed of 3 injections. The average of the replicates is used for reporting.

If referencing SW846 9060, quadruplicate analysis is required for all samples. Each sample will be composed of 4 individual injections. Report all four individual injection readings and the average of the four readings. If the client desires to have SW846 9060 cited, but does not want quadruplicate analyses performed, the default procedure for analysis will be followed and the method citation will be changed to SW846 9060Mod.

3 Shut down instrument as explained in EN CHEM SOP, WCM-69.

QUALITY CONTROL:

Replicates
Replicates should meet a %RSD of 20 (unless the sample concentrations are < 5x the EQL). If not, repeat analysis, dilute sample and repeat, etc. until criteria is met.

Initial Calibration Verification (ICV)
The ICV must be run immediately after calibration. It must be from a second source than used for calibration and meet current control limits of ± 10% of the true value. If not, recalibrate.

Initial Calibration Blank (ICB)
The ICB must be analyzed after the ICV and be less than the absolute difference of the estimated quantitation limit (EQL). If not, recalibrate.

Method Blank (MB)
The MB is carried through all prep procedures and analyzed with a frequency of 5%. The method blank concentration must be less than the EQL. If the MB concentration is greater than the EQL, all samples will be re-analyzed unless the sample concentrations are greater than 20x the MB concentration. If MB concentration is between MDL and EQL, samples will be qualified with an A flag unless the sample concentration is greater than 20x the MB concentration. If the MB fails these criteria, analysis should be stopped, and the problem should be identified and corrected before analysis can resume.

Laboratory Control Sample (LCS)
An LCS consisting of known concentration must be prepared and analyzed for each batch of 20 samples, and must meet the current control limits of 84-110%. If LCS is outside of the control limits, the system is out of control. Terminate the analysis and identify the problem. Once problem has been corrected, analysis can be resumed.

Continuing Calibration Verification (CCV)
The CCV is analyzed after every 10 samples. It must be from a second source than used fro calibration. Rejection criteria is ± 10% of true value. If the CCV fails, the problem must be corrected and the previous 10 samples between the CCV and the last CCB must be reanalyzed.

Continuing Calibration Blank (CCB)
The CCB is analyzed after every CCV. The absolute value must be ≤ EQL. If the CCB fails, the problem must be corrected and the previous 10 samples between the last CCB and the CCV must be reanalyzed.

Matrix Spike
A spike must be performed on each group of samples of a similar matrix type with a frequency of 5%. Recovery must meet the current control limits of 75-126%. MS is prepared at 10 ppm by adding 0.10 mL of 2000 ppm stock standard to 19.6 mL sample (20 mL final volume).

each group of samples of a similar matrix type with
a current control limits for accuracy of 75-126% and
must meet the current control limits for precision of 0-

ted sample results in mg/L calculations are only necessary if a

Dilution Factor = TOC as NPOC (mg/L)

spike duplicate must be performed on each group of samples of a
with a frequency of 5% and meet the current control limits for accuracy.

SSR = Spike Sample Result
SR = Sample Result
SA = Sample Added

tion:

Recovery = $\frac{SSR - SR}{SA}$

oth spike recoveries are outside of the specified control limit, the corresponding parent
ample is to be diluted and MS/MSD run on the dilution. Dilute appropriately until an acceptable
recovery is obtained. If dilution results in sample being < EQL, then flag with [ED. If only the
matrix spike OR the matrix spike duplicate are out of control for accuracy, then the corresponding
parent sample is flagged with the [N qualifier.

If the analyte of interest is greater than the linear range, dilute appropriately and perform MS/MSD
on the sample; however, the [N qualifier is not required. Also, if the analyte of interest is greater
than 4x the level of the spike concentration, accuracy calculations are not necessary.

If there is insufficient volume available for an MS/MSD, perform an LCS/LCSDUP

PRECISION:

A matrix spike duplicate must be analyzed on each group of samples of a similar matrix type with
a frequency of 5% and meet the current control limits for precision. If method SW846 9060 is
referenced, precision is to be at a frequency of 10%.

Use relative percent difference (RPD calculation):

MS = Matrix Spike Value
MSD = Matrix Spike Duplicate Value

$$RPD = \frac{|MS-MSD|}{(MS+MSD)/2} \times 100$$

If the RPD is outside of the acceptable control limits, the reported sample result is to be qualified
with the [* flag.

If there is insufficient volume available for an MS/MSD, perform an LCS/LCSDUP.

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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.

SAFETY:

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

INSTRUMENT OPERATING PROCEDURE

INSTRUMENT TYPE:	Total Organic Carbon (TOC) Analyzer	
MANUFACTURER:	Rosemount Analytical, Dohrmann Division	
MODEL:	DC 190	
SERIAL NO.:	DC 190 Electronics Module	Serial No. 96274012
	DC 190 IR Detector Module	Serial No. N6H3681C
	DC 183 Sludge/Sediment Sampler	Serial No. 96348002
DEPARTMENT:	Inorganics - Wet Chemistry	

PROCEDURE:

The DC 190 TOC Analyzer can be configured several ways. This procedure will explain the configuration for the analysis of waters (WTOC) and soils (STOC) and will be dealt with in separate sections.

I. Waters TOC (WTOC) configuration

A. Routine Operation

1. The unit will be in a stand down mode prior to start up. The unit and the detector are always "on".

2. The fan for the detector and the combustion tube, as well as the one in the soils unit, should always be "on".
NOTE: Do not touch any of the buttons or knobs on the detector !

3. Check the waste drainage port. The waste tray should be emptied prior to and during operation (if it becomes full).

4. Check the volume of acid in the amber bottle and add if volume is 1/2 or less. (20% phosphoric acid.)

5. Check that all tubing from the previous run has been reconnected (needed only after soils runs).

6. Remove any water or moisture from the water trap (inside the unit).

7. The main screen should read:
   1. System Status
   2. Auxiliary Functions

   Press # 2, it will read # 1:Omit outliners, etc.

- Press # 5, Service Functions, it will read # 1: Prime acid.
- Press # 1 and it will read # 1: Acid to IC Chamber.
- Press # 1 and it will add acid. When it is finished, return to the main menu by pressing MAIN.

Press # 1 for System Status. Flow rate # 1 and furnace set point # 4 will be shown. Turn on O_2 valves in this order ONLY: Open the tank valve, then SLOWLY open the regulator valve. Doing the reverse may blow out the valves!

The display pad has two buttons - one labelled Furnace and the other Carrier gas. Press the Carrier gas button. The light will change from red to green. Press the furnace button. The light will change from red to green when the furnace temperature has reached the set point. Analysis cannot begin until the set furnace temperature has been reached. The temperature will be 550 °C at stand down, and should be set at 680 °C for analysis.

The flow rate should be adjusted to 200 mL/min $\pm$ 20 %. This can be adjusted at the regulator.

8. Change the rinse water container DAILY. This should be acidified Milli-Q water. The sparge arm assembly can be manually moved out of the way (to remove the sample tray). The sample arm will not move manually. Follow the flow diagrams on pg. 2-19 of the manual to move the sample arm.

9. Check PEEK tubing (the beige tube on sample arm and sparge arm) to be sure it is clean and straight.

10. Observe the baseline (bottom right corner of display screen).

11. The unit is now ready to start analysis for all aqueous modes. Except POC: lithium hydroxide must be added to the scrubber tube seen as # 7 on pg. 2-7.

B. For ASM/NPOC:

1. Set parameters prior to beginning analysis. Check parameters even if the previous run was similar. When analysis is started, the parameters are locked in. To change them after analysis has begun, press start/stop 5 times to terminate the run.

   Press the NPOC button. A list of actions will appear.
   - Injection volume
   - Number of repeats
   - Sparge time
   - Acid volume
   - Rinse or stir
   - Print set up

a. Injection Volume can range from 10 µL - 400 µL. For most water, it should be set to 400 µL. If the autorange is on, this volume will change according to what the TOC reads. To change the volume prior to analysis, press # 1 to highlight the volume to the right. Press Clear. Type in the desired volume and press Enter.

b. Number of Repeats corresponds to the number replicates of a single vial. The default is three for all vials to be read.

c. Sparge Time refers to how long a sample will be sparged with O_2. The default is three minutes.

d. Acid Volume corresponds to the amount of acid added when sparging: one pulse is equal to 100 µL.

e. Rinse or Stir refers to another menu, which lists the number of rinses with water, the number of rinses with sample, stir time (stirring prior to each aliquot of sample or standard), autorange (yes or no), and CG on or off at the end of a run.

If there are any changes made to this set up, it should be noted prior to the run and noted in the run log.

2. When beginning a first run for the day, determine a new calibration factor and system blank. After these have been set, any number of sets of 32 vials can be run with that calibration for that day. The same calibration may be used on consectutive days as long as the calibration check standards pass to demonstrate the instrument is still in calibration..

Press the calibration button. A list of actions will appear.

- The calibration factor shown will be from the previous day's set up.
- The system blank shown will be from the previous day's set up.
- The sample size as set from NPOC mode.
- Standard concentration
- Update calibration factor
- Update system blank
- Other actions

Press # 1 and the value will be highlighted. Press clear, type in 1.0, press Enter.
Press # 2. Press clear, type 0, press Enter.
4: Make sure this is the value that will be read.
(Numbers 5 and 6 are used when in Boat/Soils mode.)

3. Note the baseline on the screen (bottom right corner). It should be steady and not drifting up or down. Once a run has started, the unit will return to the memorized baseline. If the baseline is unsteady during the run, the results will also show drift,

or there will be a time out error. Check the manual's troubleshooting section if this occurs.

C. Begin analysis

1. Sample vials should be 1/2 to 2/3 full. Any more than this will result in carryover from one vial to another via the orange rubber septum.

2. Place the black tray onto its peg, line up the silver push peg with its slot on the tray. Try not to force the tray to turn, it has memory regarding each vial position. Make sure samples and standards are well mixed prior to aliquoting.

D. Calibration

Check manual for method-specific calibration.
To calibrate ASM/NPOC : check parameters.
In vial # 1, place the standard
In vial # 2, place the same standard with a peg on the outside of the circle.
In vial # 3, place Milli-Q water
In vial # 4, place Milli-Q with a peg on the inside of the circle
The unit will automatically perform the calibrations. Follow with calibration checks and LCS or samples. A CCV/CCB set should follow every 10 vials. Keep track of vials on the run log. To terminate the analysis, place a peg on the outside circle next to the last vial to be read.

If at any point the analysis needs to be terminated, press the start/stop button 5 times.

When the analysis is complete, send the information to the computer and printer.

Another method can now begin or the unit can be shut down.

E. Shutdown

Leave the furnace "on" at all times, the exception being if it is not to be used for several weeks. If it is not to be used for several days, go into the system menu (from the main menu) and change the set temperature for the furnace to 550 °C.

Turn the carrier gas off. Turn the regulator valve off, although the tank can be left on.

Empty vials. Dump the waste tray.

DO NOT turn either the detector or the unit itself off.

II. Soils TOC (STOC) Configuration

A. Routine Operation

Beginning with the unit in a shut down mode:

1. Open the door and take the teflon tube out of the copper side of the tin/copper scrubber. Note its position prior to removing (ie., how much tube is actually inside the U tube). Connect the tube labelled soils (with black shrink tubing around it) and connect it to the tube from the copper side.

2. Place the tube and septa into the sparger on the soils unit.
NOTE: Remove after analyzing soils - H_2O will travel back into the furnace if this is not done.

3. Turn boat gas and sparge gas on (remember to turn off when done!).

4. Turn power on (black switch). Indicator light shows when it has warmed up.

5. Turn the carrier gas on at the tank, then at the regulator. In the NPOC/Boat mode, type in the concentration units: (µg/g or mg/L). For solids (soils), use µg/g. Sample size indicates size of standards to use. Analysis can now begin.

   NOTE: In this mode, after reading the boat, the unit will ask if you want to continue (meaning: Do you want to perform more than one replicate?). Generally, as with waters, a sample or standard can be read in triplicate and the first value omitted. In all modes but the ASM/RSM, this can be done by the TOC itself. See Omitting an outliner.

6. Prepare samples as described in the SOP for soils. A homogeneous mix should be acheived. Have the sample or the standard ready to inject: that is, weighed or aliquoted, in the boat, the boat in the carrier with the lid closed, prior to pressing the Start button.

B. Calibration:

1. Select the calibration function and change the necessary items (ie., calibration standard concentration).

2. In order to acheive a mean value for any standard, perform at least three injections. After this, the unit asks to continue or not, press no. The unit will perform the calculations. Omit any numbers, if needed, at this time.

3. Select the calibration function and update the calibration factor. Since there is no blank to be standardized, a system blank need not be updated.

4. Calibrate with the standards used in the SOP for the Boat mode. When complete, the unit is ready to read samples. Apply the same procedure for the soil samples. Load the boat and place in the slide. Press Start.
5. Follow the guidelines for MS/MSD and other qualifications as stated in the SOP for soils analysis.

C. Shut down

1. Remove boat from slide.

2. Turn the boat gas and sparge gas off. Turn the carrier gas off if no further analysis of waters is needed. Turn both the tank and the regulator off.

3. Remove the tube from the gas sparger tube on the soils unit.

4. Turn the power off on the soils unit (black switch).

5. Open the door of the autosampler unit and remove the tube from the outer black shrink tubing. Place the tube (without black tubing) into the copper side of the tin/copper scrubber. Return the tube in the copper scrubber to its position prior to removal. This is important for gas circulation in the tube.

6. The fan for the soils unit will always be on in order to cool the furnace.

III. Routine/Preventative Maintenance

Check the manual for troubleshooting and any routine maintenance.

REVIEWED BY: Jeffrey A. Gordon — 4-26-99
Jeffrey A. Gordon
Inorganic Section Supervisor
Date

Gregory J. Graf — 4-26-99
Gregory J. Graf
Quality Control Officer
Date

APPROVED BY: Glen A. Coder — 4-27-99
Glen A. Coder
Laboratory Manager
Date

APPENDIX G.

MPCA SAMPLING STANDARD OPERATING PROCEDURE

FINAL

Superfund and Voluntary Investigation and Cleanup Programs Minnesota Pollution Control Agency

Sampling Standard Operating Procedure at Anoka County Park

Pilot-Scale Study for Enhanced *in-situ* Bioremediation of Chlorinated Solvents via Vegetable Oil Injection

Rev. 2; Dated 08/30/01

(Approved by MPCA on 08/30/01)

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Ref. 1: Final Work Plan for Field Application To Enhance *In-Situ* Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection a the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota, May 2001 and any updates thereto, prepared by Parsons Engineering Science, Inc.

Superfund and Voluntary Investigation and Cleanup Programs

Sampling Standard Operating Procedure at Anoka County Park

Pilot-Scale Study for Enhanced *in-situ* Bioremediation of Chlorinated Solvents via Vegetable Oil Injection

1.0 INTRODUCTION

This document outlines procedures to be used for ground water quality measurements and for collecting and handling ground water samples obtained from monitoring wells at Anoka County Park (ACP) near the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota during the pilot-scale study for enhanced *in-situ* bioremediation of chlorinated solvents via vegetable oil injection. Deviations from these procedures may be required by unforeseen circumstances that develop during the sampling event(s). Such deviations will be approved by the lead technical staff or the field crew leader as described below. When regulatory or lead technical staff approvals cannot be obtained in advance, deviations from the established procedures will be evaluated as soon as possible after sampling and the need for re-sampling will be evaluated. Deviations from the specified procedures will be clearly noted on the sampling information form (SIF) used for the sampling of each well and will be included in the Sampling and Analysis Report.

This document will be used in conjunction with the *Final Work Plan for Field Application To Enhance In-Situ Bioremediation of Chlorinated Solvents Via Vegetable Oil Injection a the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, Minnesota, May 2001* and any updates thereto, prepared by Parsons Engineering Science, Inc. [Ref. 1].

2.0 ADVANCE PREPARATION FOR SAMPLING

Selection of analytical parameters, laboratory arrangements, the order of sampling wells, field measurement and sampling techniques, equipment selection and other quality assurance measures are based on the sampling objectives presented in the main body of the Sampling and Analysis Plan.

2.1 Selection Of Analytical Parameters

Samples will be collected for analysis of the parameters shown in Table 4.4 of Ref. 1 to fulfill requirements of the MPCA Superfund and/or Voluntary Investigation and Cleanup (VIC) programs. Samples will be collected from the wells and analyzed for the parameters as listed in Tables 4.1, 4.2, and 4.3 of Ref. 1.

Analytical techniques for organic compounds were selected primarily on the basis of ability to detect potential contaminants at low levels.

2.2 Detection Limits

Practical quantitation limits are listed in Table 1.2 of the project specific QAPP (Appendix D of Ref. 1).

2.3 Quality Assurance For Field Procedures

Particular care will be exercised to avoid the following common ways in which cross contamination or background contamination may compromise ground water samples:

- improper storage or transportation of equipment

- contaminating the equipment or sample bottles on site by setting them on or near or downwind of potential contamination sources such as uncovered ground, a contaminated vehicle, or vehicle or generator exhaust
- handling bottles or equipment with dirty hands or gloves
- inadequate cleaning of well purging or sampling devices

Field methods quality assurance verification procedures are described below in Section 4.4, "Field Blanks, Replicates and Split Samples". Field personnel should work under the assumption that contamination exists in land surface, soil and vegetation near sampling points, wash water, etc. Therefore, exposure to these media will be minimized by taking at least the following precautions:

- minimizing the amount of rinse water left on washed materials
- minimizing the time sampling containers are exposed to airborne dust or volatile contaminants in ambient air
- placing equipment on clean, ground-covering materials instead of on the land surface

Clean gloves made of appropriately inert material will be worn by all field crew. Gloves will be kept clean while handling sampling-related materials. The gloves will be replaced by a new pair between each sampling site.

2.4 Sampling Containers And Preservatives

Laboratory-supplied sampling containers and preservatives to be used for samples from all wells are shown in Appendix B of Ref. 1. The Laboratory Quality Assurance Project Plan (Lab-QAPjP) (Appendix F of Ref. 1) includes specific procedures for the following: sample container cleaning, testing, labeling and storage; preparation and addition of preservatives. Preservatives for volatile organic samples are added to the sample container in the field. Chemical preservatives for all other parameters are added in the laboratory before samples are collected.

2.5 Purging And Sampling Equipment

Well purging and sampling equipment includes the following:

- decontaminated submersible pumps and/or bladder pumps
- decontaminated pump discharge lines
- other equipment such as rope, gloves, generators, air compressors (with air/oil filter), etc.

2.6 Decontamination, Storage And Transport Of Equipment

Decontaminated pump tubing will be used each time each well is sampled. Between sampling events, the tubing will be stored in a sealed, chemically inert plastic bag. Pump bladders will be decontaminated by circulating decontamination fluids through the pump as described below.

All sampling-related equipment including filtration devices, personal protection gear and materials coming into contact with actual sampling equipment or with sampling personnel will be decontaminated. Decontamination will be performed before and after working at each sampling point, at a decontamination station in the field or at each individual sampling point in the field. All equipment will be handled in a manner that will minimize cross-contamination between wells and avoid introducing surface contamination or ambient air contamination into a well.

Before mobilizing for field work or performing any decontamination, a source of "control" water and organic-free deionized water for decontamination will be selected and evaluated. The evaluation process will include sufficient laboratory analysis to assess the suitability of the proposed water. The proposed decontamination water will only be used for decontamination if analyses indicate it is appropriate for the complete set of target parameters. In the event that use of a desorbing agent is necessary, the desorbing agent will be reagent grade isopropyl alcohol . Equipment will be decontaminated in the following manner:

A. Equipment that does <u>not</u> contact sample water or the inside of the well:

- clean (inside and out) with an Alconox/clean-water solution - applied with a scrub brush where practical
- rinse with clean "control" water
- inspect for remaining particles or surface film and repeat cleaning and rinse procedures if necessary

B. Equipment that contacts sample water or the inside of the well:

- clean (inside and out where possible) with an Alconox/clean-water solution - applied with a scrub brush made of inert materials
- rinse with potable water
- rinse with isopropyl alcohol
- rinse with organic-free deionized water
- inspect for remaining particles or surface film and repeat cleaning and rinse procedures if necessary
- shake off remaining water and allow to air dry

The internal surfaces of pumps and tubing that cannot be adequately cleaned by the above methods alone will also be cleaned by circulating decontamination fluids through them. The fluids will be circulated through this equipment in the order shown above under "B".

Wastewater from well purging and equipment cleaning will be containerized on-site until analytical results are obtained to determine proper disposal. Disposable personal protective and sampling equipment will be containerized on-site and staged inside NIROP building for disposal at a sanitary landfill after characterization.

When transporting or storing equipment after cleaning, the equipment will be protected in a manner that minimizes the potential for contamination. The tubing will be placed in a clean, inert plastic bag or wrapped in aluminum foil.

2.7 Selection Of Sample Collection Techniques

Sample collection techniques outlined in this document have been tailored to the goals of this sampling event and the individual characteristics of this site. A summary of the sampling goals and the pertinent site, well and contaminant characteristics is given in the Sampling and Analysis Plan.

2.8 Order of Sampling

The ground water monitoring wells will be purged and sampled in the following order: contingency wells, monitoring wells (starting from the most downgradient well), injection wells, and background well to minimize cross contamination.

3.0 PRELIMINARY FIELD WORK

3.1 Field Inspections And Field Decisions

Before purging or sampling, all wells should be inspected to verify that:

- all sampling points are safely accessible;
- all wells are in satisfactory condition;
- current water levels indicate a gradient consistent with the preliminary order of well sampling;
- the existing health and safety plan procedures are appropriate for actual site conditions;
- well depth and that the annular seal is intact at the surface.

Any unusual conditions including the presence of wind-blown dust or odor in the ambient air should be recorded on the SIF.

3.2 Detection Of Immiscible Layers

Air inside a well suspected of significant contamination will be tested immediately with an organic vapor detecting device such as PID or FID. The measurement will be recorded on the SIF. If immiscible layers of contaminants (free product "floaters" or "sinkers") are suspected or if odors or an oil sheen are observed, procedures will be followed to characterize the distribution of contaminants in the water-yielding zone adjacent to the well screen. Because free product can accumulate anywhere from the top to the bottom of the water column, the normal sequence

of purging and sampling will be preceded by a free-product evaluation step to allow for the best characterization of contamination. An attempt to measure the thickness of any free product will be made using the following equipment: electric water-level probe or oil/water interface probe. General procedures for detection and sample collection of immiscible layers will be in accordance with guidance provided in U.S. EPA RCRA Ground-Water Monitoring: Draft Technical Guidance, November 1992, Section 7.2.3; specific detailed procedures actually used in response to site/well conditions will be recorded on the SIF and included in the Sampling and Analysis Report. The presence of and characteristics of any detected immiscible layers will be noted on the SIF.

A bailer will be used to collect any pre-purging samples from the water table surface and a thief sampler will be used to collect any pre-purging discrete-interval samples from below the top of the water column. In addition to any discrete-interval samples collected, an additional sample will be collected from near the middle of the water column after normal purging. Analytical needs for these three samples will be reviewed with the Superfund Program technical representative to determine which analyses are required for each sample. Visual screening or sequential analysis of samples may eliminate the need to analyze all samples collected in some circumstances.

3.3 Water-Level Measurements

Prior to any well evacuation or sampling, initial static water levels will be measured and recorded for all wells. This is done to facilitate selection of the proper pump intake depths for purging and sampling and calculation of the ground water flow direction.

During initial static water level measurement, a minimum of two water level measurements will be made at each well. The two water level measurements will be made in rapid succession. If there is poor agreement between the first and second static water level measurements (i.e., a difference of more than 0.01 feet), data will be re-evaluated for measurement errors, unsuspected pumping that may be causing transient changes in gradient, etc. If the discrepancy cannot be rectified, a third static water level measurement will be made at each questionable sampling point to assess the true water level, verify non-steady state conditions, etc.

The sampling crew will make water-level measurements at all appropriate monitoring wells and piezometers within the shortest time interval practical to provide comparable numbers by which to calculate the ground water gradient. A time limit exceeding 8 hours will be considered a reportable protocol exception for this sampling event. An additional water level measurement will be taken immediately after sampling to evaluate potential cascading problems. These water levels will be entered on the SIF.

Water levels will be measured with an electric water-level probe. The depth-to-water should be referenced to the measuring point marked at the top of the innermost well casing. Where a measuring point has not been marked at the top of the casing, the measuring point will be assumed to be at the top of the innermost casing on the north side of the casing. When reporting absolute water level elevation, this measurement will be converted to water level elevation (MSL) from the surveyed elevation of the top of well casing. Water level measurement data will be recorded on the SIF.

3.4 Field Water-Quality Measurements

Specific conductance, pH, temperature, turbidity, dissolved oxygen, and oxidation/reduction potential will be measured in the field immediately before sample collection. All measurements will be recorded on SIF. Purging and stabilization information will also be noted on the SIF.

All measurements except for turbidity will be taken within a closed flow cell device designed to allow measurement of these parameters while minimizing changes in temperature, pressure, and dissolved gases from the in-situ aquifer environment. The flow cell has the following characteristics:

- Air tight fittings for installation of all probes.
- Intake is connected directly to the pump discharge line.
- Resides in a water bath kept at a temperature close to the in-situ ground water temperature.
- A discharge line of sufficient length that is connected to the flow cell with an air tight connection.

- A maximum volume of no greater than five times the per minute volumetric rate of inflow to the cell to maintain measurement sensitivity to temporal changes in water quality.
- A minimum volume of 350 ml to provide enough thermal mass to minimize external temperature effects.
- The flow cell will be shielded from strong winds and on hot days it will be shielded from direct sunlight.

The operation of the probes will be as follows:

1. The flow of extracted ground water through the flow cell will be maintained as continuous and steady as practical throughout the measurement period.
2. Discharge rates through the flow cell are kept low enough to prevent streaming potential problems with probes.
3. All probes will be fully immersed without touching the sides of the air tight, non-metallic flow cell.
4. All probes will be allowed to equilibrate with fresh well water for a minimum of five minutes before recording measurements.

Specific procedural details for measurement of individual field water quality parameters are outlined in the manufacturer's instruction/owner's manual. General care, maintenance, calibration procedures, and operation of each measurement device will also follow manufacturer's specifications as detailed in the instruction/owner's manual for each device.

3.5 Purging And Stabilization

Before a well is sampled for the dissolved phase, it will be purged as described above to ensure that samples contain fresh formation water. While the well is being purged, water quality parameters described above in Section 3.4, "Field Water-Quality Measurements", and the quantity of water purged will be recorded on the SIF.

A purging rate that will minimize drawdown while allowing the well to be purged in a reasonable length of time will be used and recorded on SIF. Care will be taken to avoid any significant amount of cascading or turbulence in the well.

Wells with extremely slow recharge rates due to tight formation materials, will require alternate purging and sampling methods. If normal purging is clearly impractical, the well will be pumped to near dryness and allowed to partially recover for a maximum of one hour. Sampling will then commence as soon as possible after evacuation.

Wells that do not have extremely slow recharge rates will be purged and sampled as described below. Purging will be conducted in a manner that, to the extent practical, removes all the "old" water in the well so it is replaced by fresh ground water from outside the well installation. Wells will be purged at a maximum purge rate not to exceed the draw down of more than two (2) feet.

1. The well will be purged by placing the pump just below the water table. (NOTE: It is possible that for some shallow wells that the water level will be within the screen. If this is the case, the same procedure for purging and sampling can be used although in some cases the pump will have to be within the screened interval due to necessity.)
2. Repeated vertical adjustment of the purging equipment intake will be avoided if possible, but may be necessary as the water level drops.
3. Decontaminated submersible pumps and/or bladder pumps will be used for purging and sampling.
4. Sampling will immediately follow purging and stabilization .

Field water quality parameters will be measured for stabilization after each water-column volume is purged. The following target criteria for three consecutive measurements (one water-column volume apart) will be used to demonstrate stabilization:

- pH +/- 0.1 units
- temperature +/- 1.0 degrees Celsius
- specific conductance (temperature corrected EC) +/- 5%
- dissolved oxygen +/-2%

- turbidity: less than or equal to 5 NTU

Samples for laboratory analysis will be collected only after a minimum of 3 water-column volumes have been purged and stabilization of field water-quality parameters has been demonstrated by meeting the target criteria defined in the preceding paragraph. If field parameters do not stabilize after approximately five water-column volumes, then field staff will check operator procedures, equipment functioning and well construction information for potential problems. In particular, field staff will re-evaluate whether or not water is being withdrawn from the appropriate depth to effectively evacuate the well.

If all the checks produce no new insight, a decision might be made to collect samples after five or more water-column volumes have been purged even if field measurements have not stabilized. Before authorizing the laboratory to analyze samples, the meaningfulness and value of completing laboratory analysis of the sampling suite will be evaluated by reviewing the results of field measurements, well construction data, site hydrogeology, etc. Where such data is presented, it will be clearly documented that stabilization was not achieved; at a minimum, this fact will be reported on the SIF and in the Sampling and Analysis Report.

As with water from well development, purge water will be properly stored, tested, and disposed of in accordance with all applicable rules including Minnesota Rule 7060. Fifty-five gallon drums will be located at each of the wells to collect water removed from the wells during development and sampling. No significant amount of well water will be emptied or discharged onto the ground surface unless analytical data are available and indicate that the water is not contaminated. After water analyses become available, and appropriate disposal alternatives are evaluated, the water will be disposed of in an environmentally safe manner that does not conflict with any applicable rules.

4.0 SAMPLE COLLECTION

This section describes procedures for setting the sampling pump and collecting ground water samples. Field data for these items will be recorded on the SIF for each sampling point.

4.1 Pump Setting

A bladder pump will be used as the default device for sample collection. If well recovery is so slow that a satisfactory water column height (for normal pump operation) is not reached in a reasonable amount of time, a Teflon® bailer will be used for sample collection. The SIF will show what type of device was used to sample each well. If any device other than the one described above is used, it should be reported as a protocol exception.

In very slowly recharging wells, the pump intake will be set approximately two feet from the bottom of the well to minimize aeration problems. Alternately, in wells where the entire screen length is saturated, the pump intake will be set in the middle of the screened interval.

The groundwater sampling pump will not be removed between well purging and groundwater sampling. Pumping will be continuous and sampling will immediately follow purging. If pumping is not continuous it will be noted on the SIF. The sample collection pumping rate will be less than or equal to the purging rate. The sampling rate will be between 100 and 250 ml/minute.

4.2 Sample Filtration

Sample filtration is not anticipated for this project. If filtration is required, MPCA and USEPA approval will be obtained prior to sampling.

4.3 Filling Sample Containers

Table 1 summarizes the sample container type, filling method, preservation method and holding time for each analytical parameter set. Individually prepared bottles will not be opened until they are to be filled with water samples.

1. A clean and dry sheet of relatively inert plastic shall be placed on the ground surface in the wellhead area. If materials used in the sampling process must be put down, they will be placed on a clean portion of the plastic sheet instead of the ground surface.
2. A clean pair of nitrile or latex gloves will be put on at the onset of sampling activities at each new sampling point.
3. Sampling personnel will keep their hands as clean as practical and replace gloves if they become soiled while performing sampling activities.

Bottles will be labeled and chain-of-custody sections will be filled out by the field personnel according to procedures described below in Section 5: "Documentation of Sampling Event". To prevent a mix up with sample bottle identification, no sampling-point specific information such as "well name" will be filled out in advance of sampling. Chain of custody information will be completed before leaving the sampling point. Laboratory-prepared bottles will be used to assure quality control.

The order of filling bottles with water to be analyzed will be as follows:

1. volatile organic compounds
2. methane, ethene, ethane
3. sample volume for mobile lab analyses
4. nitrate and nitrite
5. total organic carbon
6. bromide

This order will be reversed in very slowly recharging wells and will be noted on the SIF. Replicate samples will be collected sequentially as described in Section 4.4: "Field Blanks, Replicates and Split Samples". Methods for filling sample containers for individual analyses are described in Table 1.

The sample water discharge point at the end of the tube will be held as close as possible to the sample container without allowing the sample tubing to contact the container. The exception to this rule is for dissolved oxygen and chemical oxygen demand samples where the container is filled from the bottom up by inserting the tube into the bottom of the container. At a minimum, sampling personnel will use their body to shield the sampling container from wind and airborne dust while filling. When strong winds, heavy rain, or dusty conditions are present, additional measures will be implemented to guard against background interference.

4.4 Field Blanks and Replicates

Sample blanks will be collected to detect background or method contamination. Replicate samples will be collected to evaluate variability in analytical methods. QA/QC samples will be collected at sampling points suspected to have relatively higher levels of contamination to provide meaningful information for blank or duplicate sample evaluation. Field duplicate samples will be assigned identification aliases on the sample bottle label and on the chain of custody sheet to avoid alerting laboratories that the sample is a replicate sample. The true identity of the field duplicate samples will be recorded in the field sampling log.

The collection schedule for QA/QC samples will be as follows:

1. one trip blank (composed of three replicate vials) for each cooler of VOC samples
2. one field methods (equipment) blank each day by each field sampling crew
3. at least one replicate set for every 10 samples collected

Field ambient air blanks will not be required for this project because the project site is a community park and no VOCs will be emitted from an operating facility during sampling. Also, no automobile engines will run during the sampling event. If the pumps use a gas-driven generator, the generator will be located downwind from the sampling point.

For each type of QA/QC sample, containers will be prepared and submitted for the analyses listed in Table 5.1 of Ref. 1.

Field Blank Samples

Methods that will be used for preparing field blank samples are described below.

Trip blanks for VOCs consist of a set of three pre-filled 40 ml purge and trap vials that will be filled and sealed by the primary VOC analytical laboratory with laboratory-controlled organic-free water. The 40 ml, purge and trap, blank sample vials will travel with the actual sample vials to and from the field in the cooler, to the well head, etc., so that the blanks are exposed to precisely the same conditions as the actual samples. The bottle blanks will not be opened until they are analyzed in the laboratory along with the actual VOC samples they have accompanied.

Field equipment/methods blanks will be collected in the field for target parameters . Sample containers used for each blank will be the same as for the actual analysis of sample water for these parameter groups. All containers shall be pre-cleaned within the laboratory's QA/QC program in the same manner as primary sample bottles. The sample blank containers will be filled in the field. Laboratory-controlled organic-free water will be used to fill all organic blank samples. Trace metals blanks will be filled with laboratory-prepared, triply distilled water. The same preservatives will be added to both the methods blank and the primary samples.

Collection of field equipment/methods blank samples will be conducted to simulate actual field sampling methods in a manner that would detect the presence of background or cross-contamination of samples from the ambient environment, preservatives or sampling equipment. An effort will be made to have the blank sample water contact all the interfaces and preservatives (where applicable) that the sample water will contact. These may include the sampling mechanism, ambient air, sample container and, when applicable, tubing, filtration membranes and preservatives.

Laboratory-supplied blank water will be pumped through a freshly decontaminated reusable sampling device and directly into the appropriate, laboratory provided, sample containers. Blanks for filtered samples (only if filtered samples are collected) will be collected by passing the blank sample water through the filtration device and the same type of filters used for collecting the primary samples.

Field Replicate Samples

Field replicate samples of actual ground water will be collected for the following parameters listed in Table 5.1 of Ref. 1. One field replicate sample set will be collected for every ten primary sampling sets. The replicate samples will be collected by sequentially filling all containers as close together in time as practical with a sampling stream that is as steady and continuous as practical. The sequence number (first, second, etc.) and time of sample filling will be listed in the field notebook. The time that each individual container was filled will be listed on the container and on the Sample Identification - field chain of Custody Record (SI-FCCR) in the same manner as primary samples. All samples will be sent to the laboratory specified in Appendix D of Ref. 1.

Field Split Samples

Field split samples are not anticipated for this project. If split samples are collected in the future, MPCA and USEPA approval of the other laboratory QAPP will be obtained prior to sampling.

5.0 DOCUMENTATION OF SAMPLING EVENT

This sampling protocol template includes the use of forms shown in Appendix B; they are designed for documentation of field activities and collection of field data. They also provide a means to verify whether or not this protocol was followed during a number of key steps in the ground water sampling event. The forms include the following:

1. Sampling Information Form
2. Purging and Stabilization Form
3. Identification - Field Chain of Custody Record (SI-FCCR)

5.1 Sample Identification

The Sample Identification - Field Chain of Custody Record (SI-FCCR) in Appendix B will be completed as described above in Section 5.0, "Documentation of Sampling Event".

The SI-FCCR will be at least a two-part (carbonless copy) form.

Each sample container will be labeled with the following information:

- unique container ID #
- sample collection Date and Time
- initials of person collecting sample
- analyses required
- preservation method

Container information will be entered at the sampling point at the time of sample collection. However, for containers receiving preservatives in advance, "analyses required" and "preservation method" will be entered onto labels by laboratory staff. For containers receiving preservatives in the field, "preservation method" will be entered at the time individual containers are filled.

5.2 Chain Of Custody

A chain-of-custody record (SI-FCCR) will be initiated in the field at the time of sampling; a copy will accompany each set of samples (cooler) shipped to any laboratory.

Each time responsibility for custody of the samples changes, the new and previous custodians will sign the record and denote the date and time. A copy of the signed record will be made by the receiving laboratory. The final signed SI-FCCR will be submitted with analytical results in the Sampling and Analysis Report.

Field Chain of Custody Documentation

All signatures related to sample custody will be made in indelible ink on the SI-FCCR in a timely fashion. One or more signatures will be entered to identify the person or persons who are collecting the samples. Each time the custody of a sample or group of samples is transferred, a signature, date and time will be entered to document the transfer. The signatures, date and time will be entered at the time of transfer. A sample will be considered to be in custody if it is in any one of the following states:

1. in actual physical possession
2. in view, after being in physical possession
3. in physical possession and locked up so that no one can tamper with it
4. in a secured area, restricted to authorized personnel

A secured area such as a locked storage shed or locked vehicle specified in the "comments" column, may be used for temporary storage. When using such an area, the time, date, and location of the secured area will be recorded in the "relinquished by" space. The time at which an individual regains custody will then be recorded in the "received by" space.

Chain of Custody During Shipping and Transfer of Samples

When samples are shipped, the person sealing the shipping container will enter the time, date and their signature on the SI-FCCR. The laboratory part of the SI-FCCR will be enclosed in the container; the top page (first part) will be retained for the project manager's file. A post office receipt, bill of lading, or similar document from the shipper will be retained as part of the permanent chain-of-custody documentation.

One or more custody seals will be affixed over the opening of the shipping container in a manner that precludes opening the container without breaking the seal(s). The container seal(s) will be inscribed with the signature of the person sealing the container and the date and time sealed.

The receiving laboratory will be notified in advance of chain-of-custody procedures that must be followed for a group of samples. The laboratory will be instructed to note whether or not the container seal(s) are intact and sign in the appropriate blank on the SI-FCCR at the time of receipt. They will also be instructed to keep a copy and return the original form to their client's quality assurance officer.

5.3 Field Sampling Log

A daily field log of sampling activities will be kept by the leader of the field sampling crew. At a minimum, the log will contain a record of the following items:

- list of field personnel present
- field conditions(see Section 5.5)
- description of exceptions to this protocol including specification of which samples may have been impacted by exception(s)
- For each well sampled:
  1) Well Name and unique SI-FCCR # used to identify samples,
  2) equipment used for evacuation and stabilization,
  3) date and time that purging and sampling began and ended,
  4) a list of all samples sent to each laboratory

5.4 Exceptions To Sampling Protocol

This protocol defines the procedures to be followed during this sampling event. Exceptions to this protocol will be noted on the SIF.

If there has been any potentially significant impact on sample integrity, then the potential impact for each parameter for each sample affected will be footnoted whenever the results are reported or referred to in the Sampling and Analysis Report.

5.5 Field Conditions

Field conditions during the sampling event will be recorded on the SIF. The Sampling and Analysis Report will include a statement regarding the likelihood that any unusual field conditions had a significant impact on the integrity of results. Field conditions reported will include but not be limited to the following:

- air temperature
- wind speed/direction
- precipitation/moisture at the time of the sampling event, and if known, previous days' precipitation
- ambient odors
- airborne dust

6.0 SAMPLE PRESERVATION, HANDLING AND TRANSPORT

6.1 Sample Preservation

Samples will be preserved as shown in Appendix B of Ref. 1. All chemical preservatives, added to containers in the laboratory or field will be produced and controlled within the laboratory's QA/QC program as reflected in the Lab-QAPjP (Appendix F of Ref. 1). Field supplies of preservatives and sample containers with pre-dosed preservatives will be discarded and replaced with fresh preservatives no later than 14 days after receipt from the laboratory.

All samples will be thermally preserved in the field immediately after sample collection by placing the samples in an insulated ice chest containing ice. The ice chest temperature will be checked by measuring the temperature of the water within the temperature blank container and recorded upon receipt at the laboratory, to verify whether or not samples are kept refrigerated at approximately 4 degrees C.

6.2 Sample Handling And Transport

All ice chests shipped will be accompanied by an SI-FCCR form and contain a complete destination and return address on the outside of the cooler. The samples will be kept at approximately 4 degrees C during transport to laboratories. Maintain the chain-of-custody according to procedures described in Section 5.2.

Table 1: Sample Containers, Filling Method, Preservation and Holding Times

Sample Containers, Filling Method, Preservation and Holding Times are provided in Appendix B of Ref. 1.

Figure 1: Location of Sampling Points

For Location of wells to be sampled under this project refer to Figure 2.2 in Ref. 1

APPENDIX A: SELECTED ANALYTICAL PARAMETERS, METHOD NUMBERS AND REPORTING LIMITS

Analytical Parameters and Method Numbers for use on this project are provided in Table 4.4 of Ref. 1.

Practical quantitation limits are listed in Table 1.2 of the project specific QAPP (Appendix D of Ref. 1).

APPENDIX B: EXAMPLE FORMS

Sampling Information Form

Weather Conditions:
Cloud Cover_____
Wind Speed & Direction_____
Temperature:_____
Precipitation:_____

Facility ID#_____
Facility Name_____
MPCA Master ID:_____
Project Name:_____

Station ID#_____
Location:_____
Well Depth (ft. Below TOC):_____
FID/PID reading @ Wellhead:_____
Depth to Water (below TOC):_____

Sample Date:_____
Sample Time:_____
Casing Diameter_____
FID/PID Background Conc.:_____

Purge Rate:_____gpm

Well Volumes Removed Prior to Sampling_____

Gallons per Lineal Foot 2"ID=0.163, 4"ID=.0661, 6"ID=1.5, 12"ID=5.88

Sampling Method:_____Tap _____Submersible Pump _____Bailer Other (detail)_____

Pump intake or bailer set at _____ft. Below TOC.

Tubing Type:_____, New, Previously Used and Cleaned was used to collect all samples Y N

Flow Cell Used Y N Purging and Stabilization Protocol Followed Y N

Sample Appearance (describe)_____

Field Cleaning of Equipment Performed _____

Describe any deviations from Sampling Protocol_____
_____
_____

Transportation (Thermal Preservation) Type:_____

Comments:_____
_____
_____

Form Completed By:_____ Well Sampled By:_____

Purging and Stabilization Form

Facility ID#:_____ Site Name:_____ Date:_____

Well #:_____ Sampling Personnel:_____ Time:_____

Time	pH	Temp.	Cond.	Dis. O_2	Turb.	Notes

APPENDIX H.

COMMENTS-RESPONSES AND APPROVALS

PARSONS ENGINEERING SCIENCE, INC.
RESPONSE TO THOMAS BLOOM'S COMMENTS

SITE: NIROP Fridley, Minnesota

DOCUMENT: Draft Work Plan for Field Application to Enhance In-Situ Bioremediation of Chlorinated Solvents via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, MN

REVIEWER: Thomas Bloom, U.S. EPA

RESPONDENT: Prepared by Parsons Engineering Science, Inc.; submitted 04/24/01

Item	Section	Page	Line	Comment	Response
1	General			The use of low flow sampling procedures is not planned for collecting groundwater samples during the pilot study. Low flow sampling techniques are currently used at the site in other sampling programs. Therefore, it appears that the use of low flow sampling during the pilot study would help to ensure comparability between groundwater quality data collected during the pilot study and data collected during other sampling programs conducted on site, and the use of low-flow sampling should be considered during the pilot study. However, the use of more conventional groundwater sampling techniques may be necessary to collect the data required to fully evaluate the enhanced bioremediation processes occurring on site (e.g., collect sample representative of a larger volume of the aquifer). Consequently, Parsons Engineering Science should evaluate carefully the use of low flow sampling and consider which sampling approach best meets the needs of the pilot study.	Concur. To ensure compatibility between groundwater quality data collected during the pilot study and data collected during other sampling programs conducted on site, low-flow sampling will be utilized when collecting groundwater quality data. The work plan will be revised accordingly.
2	7	6-1		The Work Plan (Section 7, page 6-1) indicates that "if the concentration of VC exceeds current	The work plan will be revised to indicate that if the concentration of vinyl chloride (VC) exceeds current

				regulatory limits at the 5 contingency monitoring wells then a contingency remedy will be implemented." However, small exceedances of the regulatory limit may not require implementation of the contingency plan. Consequently, the Work Plan should be revised to indicate that if the concentration of vinyl chloride (VC) exceeds current regulatory limits at the contingency wells, the NIROP partnering team will be consulted to determine if the implementation of a contingency remedy is necessary.	regulatory limits plus existing ambient concentrations at the contingency wells, the NIROP partnering team will be consulted to determine if the implementation of a contingency remedy is necessary.
3	2.1.1	2-3		The Work Plan (Section 2.1.1, page 2-3) indicates that "a groundwater mound is present near well MW-46S in ACP," and that "this mound effectively limits the groundwater flow (and contaminant flux) to the river." This language appears to be taken from the 1999 Annual Monitoring Report (AMR). However, interpretation of the flow in Anoka County Park (ACP) has been revised in the February, 2001 Technical Memorandum for Additions to the 1999 AMR to indicate that the groundwater mound is a groundwater ridge and that the area in the vicinity of well MW-46S may be a stagnation zone. It has also been agreed that the AMR, as amended by the Technical Memorandum, would no longer indicate that the mound effectively limits the groundwater flow (and contaminant flux) to the river. To ensure that the current interpretation of the groundwater contours in ACP is accurately represented in the Work Plan, the above cited language from Section 2.1.1 of the Work Plan should be revised.	Concur. The work plan text and Figure 2.2 will be revised to reflect the interpretation of groundwater flow in Anoka County Park presented in the February 2001 Technical Memorandum for Additions to the 1999 AMR.

PARSONS ENGINEERING SCIENCE, INC. RESPONSE TO THOMAS BLOOM'S COMMENTS			
4	General	Although originally indicated during previous meetings, the injection of a bromide tracer no longer appears to be planned during the pilot study. It is understood that there is reluctance to pursue the injection of a bromide tracer because Health Department officials indicate limited previous experience with such practices. While it may not be appropriate to delay the beginning of the pilot study by seeking to include the injection of a bromide tracer, further consideration should be given to injecting a tracer at some later point in the study. The groundwater flow and contaminant migration patterns in ACP have proven to be difficult to establish with certainty. Moreover, groundwater flow patterns will likely change with the changes in the extraction system that are now in progress. Consequently, the injection of a tracer may be very useful for verifying that monitoring wells, particularly the contingency wells, are properly placed and screened to intercept groundwater passing through the treatment zone.	We agree that a tracer such as bromide would be useful for determining groundwater flow patterns in the vicinity of the pilot test. Previous experience using bromide as a tracer has shown that high concentrations of bromide are necessary to afford detection at even short distances downgradient from the injection point. It is not known with any degree of certainty what effect the influence of high concentrations of bromide would have on the microbes that facilitate biodegradation of the chlorinated ethenes at the site. In lieu of using bromide, changes in contaminant concentrations and groundwater geochemistry should be sufficient to determine where vegetable oil is influencing the groundwater system and enhancing biodegradation.

PARSONS ENGINEERING SCIENCE, INC.
RESPONSE TO DAVID DOUGLAS' COMMENTS

SITE: NIROP Fridley, Minnesota

DOCUMENT: Draft Work Plan for Field Application to Enhance In-Situ Bioremediation of Chlorinated Solvents via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, MN

REVIEWER: David Douglas, Minnesota Pollution Control Agency

RESPONDENT: Prepared by Parsons Engineering Science, Inc.; submitted 04/24/01

Item	Section	Page	Line	Comment	Response
1	General			The MPCA staff requests that the work plan be modified to reflect the data quality objectives (DQOs) agreed to in Charleston. The MPCA staff has not had time to review the statistical methods proposed by Tom Johnson of TtNUS. The MPCA staff approval of this document does not constitute approval of the proposed statistical methods or other modifications made subsequent to the Charleston meeting. A separate MPCA staff review of the latter modifications will be forthcoming.	The work plan will be revised to reflect the DQOs discussed in Charleston. The meeting minutes will be included as an Appendix.
2	2.1.1	2-3		The MPCA staff requests that the Navy remove the last sentence of this section. We have worked with the Navy to revise statements in the 1999 Annual Monitoring Report (AMR) and this sentence was removed from the 1999 AMR. Please note that significant modifications are currently being made to the pumping remedy upgradient of the pilot test area. Several additional pumping wells will be brought on line prior to the implementation of the Pilot Test. These pumping changes may make changes to the ground water flow	Concur. The work plan text and Figure 2.2 will be revised to reflect the interpretation of groundwater flow in Anoka County Park presented in the February 2001 Technical Memorandum for Additions to the 1999 AMR. As discussed at the February 1, 2001 meeting held at NIROP, and discussed in Section 4.3.1 of the work plan, "The final [vegetable oil pilot test] well layout may vary from what is shown in Figure 2 as a result of information discovered during the field program. For example, the direction of groundwater flow may be modified when the new extraction wells are placed on-line in Spring 2001, and the well layout may need to be adjusted to

				regime in the study area and may reduce the flow of volatile organic compound (VOC) contaminated ground water into the study area. The MPCA staff requests that the impact of the new pumping wells are evaluated when the data from the pilot test is evaluated.	accommodate for these changes. The vegetable oil pilot testing system will not be installed until after the new extraction wells are brought online and the effect of these wells on the groundwater flow regime is evaluated.
3	2.2	2-8		The MPCA staff requests that the Navy remove "which may be prohibiting contaminant flux to the Mississippi River thereby allowing elevated concentrations to exist" from the partial sentence at the top of the page. We have worked with the Navy to revise statements in the 1999 AMR and the concept that the mound prevents movement of contaminated ground water to the river was removed.	Concur. The work plan text and Figure 2.2 will be revised to reflect the interpretation of groundwater flow in Anoka County Park presented in the February 2001 Technical Memorandum for Additions to the 1999 AMR.
4	4.1			The MPCA staff requests that the Navy clarify whether excess oil will be removed from the injection wells after the injection phase or just sampled.	Excess oil will not be removed from the wells.
5	Table 4.1			The MPCA staff requests that the Navy include sulfate in the pre-injection sampling analyte list and consider sampling for hydrogen in the pre-injection sampling event so that the post-injection concentrations can be compared to pre-existing conditions.	Table 4-1 will be revised to add Sulfate to the pre-injection sampling analyte list. Hydrogen analyses would be very beneficial and will be added to all applicable sampling events
6	4.2			The MPCA staff requests that prior to any field work mobilization, that the Navy give Paul Estuesta, MPCA On Site Inspector, a two-week advance notice. Paul can be reached at 651-296-7997. If this is not done, fieldwork may be delayed.	Concur. Paul Estuesta, MPCA On Site Inspector, will be given at least a two-week advance notice before field work. We understand that Paul can be reached at 651-296-7997.

PARSONS ENGINEERING SCIENCE, INC. RESPONSE TO DAVID DOUGLAS' COMMENTS					
7	4.2.1			Underground utilities clearances must be obtained through our Gopher State One Call and clearance must be documented.	Concur. Underground utilities clearances will be obtained through the Gopher State One Call program and clearance will be documented.
8	4.2.3.3			**Sampling Contingencies**: Any change in well locations must be approved by MPCA/EPA.	Concur. Any significant change in well locations will be approved by MPCA/EPA.
9	4.2.4			**Waste Handling**: This activity must be done in accordance with existing protocols (see Tim Ruda of UDLP).	Waste Handling will be conducted in accordance with existing protocols. Tim Ruda of UDLP will be contacted to ascertain existing protocols
10	4.3.2	4-7		**Drilling and Soil Sampling Procedures:** The MPCA staff requests that the Navy prepare detailed lithologic logs for each of the borings in the pilot test area and cross sections or a fence diagram constructed for the final report. The MPCA staff requests that the depth to water table be indicated on each log. Any geologic conditions which may impact the ability of the uniform delivery of vegetable oil to the desired aquifer interval or otherwise effect the success of the test and the interpretation of the results should be noted and discussed in the evaluation of the pilot test data.	Concur. Detailed lithologic logs for each of the borings in the pilot test area and cross sections or a fence diagram will be constructed for the final report. Furthermore, the depth to water table will be indicated on each log. Any geologic conditions which may impact the ability of the uniform delivery of vegetable oil to the desired aquifer interval or otherwise effect the success of the test and the interpretation of the results will be noted and discussed in the evaluation of the pilot test data.
11	**4.3.3.5**			**Injection well Development**: Under Minnesota Rule 4725 a licensed well driller must develop all new wells.	Concur. A licensed well driller will develop all new wells.
12	**4.4.4**			**Onsite Groundwater Parameter Measurement.** The MPCA staff requests that the Navy include oxygen and Eh as stability criteria for ground water sampling.	Concur. Dissolved oxygen and oxidation-reduction potential will be included as stability criteria for groundwater sampling.

PARSONS ENGINEERING SCIENCE, INC. RESPONSE TO DAVID DOUGLAS' COMMENTS					
13	**4.4.2.2**			**Preparation for Sampling:** The MPCA staff requests that the Navy documents all calibration procedures of field equipment and include information in final report.	Concur. All calibration procedures of field equipment will be recorded and included in the final report.
14	4.4.4			**On-Site Ground water Parameter Measurements:** In accordance with MPCA Site Remediation Guidance Documents (Appendix 2 of 1998 Superfund and Voluntary Investigation and Cleanup Programs, Sampling Protocol Template for Monitoring Wells, Section 3.5), the MPCA staff requests that the Navy use stabilization parameters criteria listed below: pH: +/- .1 units Temperature: +/- .1 Degree C Conductivity: +/- 5% Turbidity: <= 5 NTU.	Concur. The suggested stabilization parameters will be utilized.
15	Appendix D			**Project Quantitation Limits, Laboratory Method Detection Limits and MCLs:** For the VOC analysis the compounds of major interest for the test and the detection limits for each compound are listed. The MPCA staff requests that the detection limits be low enough to meet the surface water quality standards.	As discussed during a conference call between TtNUS and MPCA on April 16, MPCA will evaluate the Veg Oil QAPP as a stand alone document. The review will be done in two phases. In Phase I, the review will focus on what is necessary for getting in the field, installing new wells, and collecting background/baseline samples. This review will be completed within two weeks. In Phase II,

PARSONS ENGINEERING SCIENCE, INC. RESPONSE TO DAVID DOUGLAS' COMMENTS					
				The QAPP indicates that samples will be sent to EnChem for analysis, although this is not the OU3 QAPP lab. In addition, Table 1.2 indicates that MDLs and PQLs for analytes are all To Be Determined, while the OU3 QAPP includes specific targets for each analyte. As verified by Venky Venkatesh to Mark Ferrey and me on April 11, 2001, based on a prior agreement at a NIROP partnering meeting that was intended to save the Navy time and money, the NIROP partnering team agreed that the OU3 QAPP would be used for this work plan. Review of the work plan by all MPCA staff reviewers indicates that the OU3 QAPP was not used for the work plan. Therefore, we are unable to approve the QAPP for the work plan because it is not consistent with the approved OU3 QAPP. The Navy may still use the OU3 QAPP for the study but it needs to reflect the parameters and DQOs of the vegetable oil pilot study.	the review will be a more extensive review of the QAPP. The resolution of these issues will be completed prior to the first progress sampling. The QAPP will be revised and re-issued at a later date based on comments from MPCA.

PARSONS ENGINEERING SCIENCE, INC.
RESPONSE TO CLIFF CASEY'S COMMENTS

SITE: NIROP Fridley, Minnesota

DOCUMENT: Draft Work Plan for Field Application to Enhance In-Situ Bioremediation of Chlorinated Solvents via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, MN

REVIEWER: Cliff Casey (SOUTHNAVFACENGCOM)

RESPONDENT: Prepared by Parsons Engineering Science, Inc.; submitted 04/24/01

Item	Section	Page	Line	Comment	Response
1	General			The Workplan should specify the collection of sediment samples from each borehole associated with monitoring wells/extraction wells. These sediments should be submitted for analysis of bioavailable iron. Our expectation is that iron reducing conditions will form downgradient and will create an environment that is more favorable for degradation of both DCE and VC. Having these samples will allow us to evaluate the variability of the iron at the site and the probability that those conditions will occur and the longevity of that iron. Samples can be sent to Cliff Casey and he will get the analyses completed.	Concur. Soil samples will be collected from each borehole associated with monitoring wells/extraction wells. These soil samples will be submitted to Cliff Casey for analysis of bioavailable iron.
2	General			Discuss the expected geochemical conditions downgradient from the injection well that are expected to occur and mechanism for degradation of VC and DCE. In other words do you expect the system will be methanogenic and thus highly reducing for reduction of these lowly chlorinated HC's or do you expect the mechanism to be oxidation under iron reducing conditions.	It is anticipated that the area in the vicinity of the vegetable oil injection wells will become methanogenic. Because of the high biologically available iron (III) concentrations, it is anticipated that the system will become iron reducing at some distance downgradient from the vegetable oil injection point.

PARSONS ENGINEERING SCIENCE, INC. RESPONSE TO CLIFF CASEY'S COMMENTS			
3	General	Specify the use of either a feed grade oil that has both nitrogen and phosphorus or the addition of nitrogen and phosphorus to the food grade oil. If you do not plan to add these micro-nutrients, discuss why they are not needed.	Food grade vegetable oil will be injected. The use of this substrate versus feed-grade vegetable oil typically requires the "jumping through" of much fewer regulatory hurdles. To date enhanced bioremediation systems of any type do not appear to be limited by nitrogen or phosphorous.
4	General	A tracer such as bromide may be helpful in elucidating the degradation of the CHC's with vegetable oil. Include the tracer in the oil or address why it is not incorporated. I should think we can provide appropriate documentation to the local health agency to alleviate any concerns they may have with use of the oil. Note, our understanding of what is happening with this pilot scale project carries great significance as it relates to full scale implementation in Anoka County Park as well as the potential to expand this effort to include injection underneath NIROP plant itself.	We agree that a tracer such as bromide would be useful for determining groundwater flow patterns in the vicinity of the pilot test. Previous experience using bromide as a tracer has shown that high concentrations of bromide are necessary to afford detection at even short distances downgradient from the injection point. It is not known with any degree of certainty what effect the influence of high concentrations of bromide would have on the microbes that facilitate biodegradation of the chlorinated ethenes at the site. In lieu of using bromide, changes in contaminant concentrations and groundwater geochemistry should be sufficient to determine where vegetable oil is influencing the groundwater system and enhancing biodegradation.
5	General	Collect geophysical gamma logs for each well that is installed. This will assist in our interpretation of the hydraulic and potential retardation conditions at the site.	The use of gamma ray geophysical logs will be specified in the work plan.

PARSONS ENGINEERING SCIENCE, INC. RESPONSE TO CLIFF CASEY'S COMMENTS			
6	General	Collect isotherm sorption data. We are interested in the importance of sorption as well as matrix diffusion in CHC's at this site. Use sediments from each monitoring /injection well installed. With the idea that some wells with silts, some with clayey components and some with sand & gravel will be tested. This should give us some idea of variation at the site. We want to run some predictive analyses at other locations at NIROP as well as this location and need data to support assumptions associated with those data needs.	Isotherm sorption tests will be conducted on one sample from the screened interval of each newly installed well.
7	General	Provide a data sheet on the vegetable oil including composition, solubility and density.	A data sheet on vegetable oil including composition, solubility and density will be added to the work plan as an appendix.
8	General	Collect bulk density of sediments at each monitoring/injection well from the screened interval.	The bulk density of soils at each monitoring/injection well will be analyzed. Samples will be collected from the screened interval.
9	General	Analyze sediment at each monitoring/injection well for fraction of organic carbon from the screened interval.	The fraction of organic carbon at each monitoring/injection well will be analyzed. Samples will be collected from the screened interval.

PARSONS ENGINEERING SCIENCE, INC.
RESPONSE TO TTNUS (MARK SLADIC) COMMENTS

SITE: NIROP Fridley, Minnesota

DOCUMENT: Draft Work Plan for Field Application to Enhance In-Situ Bioremediation of Chlorinated Solvents via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, MN

REVIEWER: Mark Sladic (TTNUS)

RESPONDENT: Prepared by Parsons Engineering Science, Inc.; submitted 04/24/01

Item	Section	Page	Line	Comment	Response
1	General			Along non-technical lines, note that naming new wells beginning with MW-1 (at a site with over 150 existing wells) may be a problem in the future. We'd suggest one of two things to address this: 1.Name the new wells in the same sequence that is currently in use (i.e., the new wells should begin with MS-55S, then MS-56S, etc). 2. Use a specific designation to this project (e.g., VG-01, VG-02).	Concur. Newly installed wells will be labeled using the following convention: Injection wells will be labeled VG-INJ-1, etc Monitoring wells will be labeled VG-MW-1, etc. Contingency wells will be labeled VG-CW-1, etc.

PARSONS ENGINEERING SCIENCE, INC.
RESPONSE TO JOEL SANDER'S COMMENTS

SITE: NIROP Fridley, Minnesota

DOCUMENT: Draft Work Plan for Field Application to Enhance In-Situ Bioremediation of Chlorinated Solvents via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, MN

REVIEWER: Joel Sanders (SOUTHNAVFACENGCOM)

RESPONDENT: Prepared by Parsons Engineering Science, Inc.; submitted 04/24/01

Item	Section	Page	Line	Comment	Response
1	4.2.4			Section 4.2.4 Waste Handling Under normal conditions, purge water is stored in the poly tanks and then pumped into the equalization tank at Bldgs 52/53 via CH2MHILL/BAYWEST. Without knowing what minor quantities of vege oil may do to the air strippers, it is probably best to keep purge water w/ oil separate drum it.	Concur. Purge water containing vegetable oil will be contained in a separate container.
2	General			There is no discussion on the bioavailibilty of Fe 3 in sediments being measured when taking split spoon samples. Cliff/Todd/Chapelle may want to include this information/sampling.	Soil samples will be collected from each borehole associated with monitoring wells/extraction wells. These soil samples will be submitted to Cliff Casey for analysis of bioavailable iron.
3	2			Section 2 - KEITH HENN - Please make sure that any revisions from the Tech Memo are corrected in section 2 i.e. mound = ridge, etc.	The work plan text and Figure 2.2 will be revised to reflect the interpretation of groundwater flow in Anoka County Park presented in the February 2001 Technical Memorandum for Additions to the 1999 AMR.

PARSONS ENGINEERING SCIENCE, INC. RESPONSE TO JOEL SANDER'S COMMENTS					
4	General			In the exec summary letter to Anoka, you mentioned Bromine injection to measure zone of influence but it is not mentioned in the Work plan unless I missed it.	We agree that a tracer such as bromide would be useful for determining groundwater flow patterns in the vicinity of the pilot test. Previous experience using bromide as a tracer has shown that high concentrations of bromide are necessary to afford detection at even short distances downgradient from the injection point. It is not known with any degree of certainty what effect the influence of high concentrations of bromide would have on the microbes that facilitate biodegradation of the chlorinated ethenes at the site. In lieu of using bromide, changes in contaminant concentrations and groundwater geochemistry should be sufficient to determine where vegetable oil is influencing the groundwater system and enhancing biodegradation.
5	4.2.1, 4.2.3.1, 4.3.3.6, 4.3.3.1	Various		Sections 4.2.1, 4.2.3.1, 4.3.3.6, 4.3.3.1 VENKY- Please help Mary out with these sections in regards to NIROP POC. NIROP POC does not really exist and field decisions/help will have to come mainly from the CH2MHILL Field/QC person (equivalent to Chris or John). The CH2MHILL Field/QC person can help with dealing w/ UDLP if necessary(Tim Ruda). I am not sure if UDLP would have any utility drawings in Anoka City Park. May have to check w/ Jeff Perry.	Section 4.2.1 will be revised as follows: **Assign Accumulation Points.** Any purge fluids and decontamination rinsate/solvents or drill cuttings generated during site work will be properly contained as specified in the Waste Management Plan (CH2M Hill Constructors, Inc, 2000a). The location for the storage of containerized waste at NIROP will be coordinated through UDLP. Waste handling procedures are outlined in Section 4.2.4. **Underground Utility Clearances.** Before any field work is conducted, each proposed intrusive sampling location at ACP will be checked for underground utilities. Underground utilities clearances will be obtained through the Gopher State One Call program and clearance will be documented. Any available utility maps

					will be obtained from Anoka County Park and kept at the site where drilling work will be conducted. **Badge and Vehicle Passes.** Personnel badges and vehicle passes will be issued as necessary for field personnel to access equipment staging and decontamination areas at NIROP. Section 4.2.3.1 will be revised as follows: Anticipated support needs are outlined in Sections 4.2.1 and 4.2.2. In the event that site access difficulties arise, Anoka County Park (ACP) personnel will be contacted to resolve the problem. The site manager and field team leader for CH2M Hill Constructors, Inc. will be responsible for notifying ACP personnel of access or coordination difficulties and working with ACP personnel to rectify any problems that may arise. First paragraph of Section 4.2.3.4 will be deleted. Second paragraph of Section 4.3.3.1 will be revised as follows: Water to be used in well installation and equipment cleaning will be obtained by the well drilling Subcontractor from an off-site source. The field scientist will make the final determination as to the suitability of water for these activities. Last sentence of first paragraph of Section 4.3.3.6 will be revised as follows: All rinsate will be collected in portable tanks or 55-gallon drums and will be staged in a designated location at NIROP. The location for the storage of containerized waste at NIROP will be

					coordinated through UDLP. Second Paragraph of Section 4.3.3.6 will be revised as follows: Potable water to be used in equipment cleaning, decontamination, or grouting will be obtained from an offsite water source by the well drilling Subcontractor. Precautions will be taken to minimize any impact to the surrounding area that might result from decontamination operations.
6	4.4.3.3 and 4			Section 4.4.3.3 and 4 KEITH HENN - Can you make sure the purging method is consistent with the Lower flow sampling method we used in October 2000 or does it matter. I would think we would want to be consistent. FYI - well 46s had 18,000 ppb tce in 1999 and 12,000 ppb tce in Oct 2000 probably due to difference in sampling methods.	Concur. To ensure compatibility between groundwater quality data collected during the pilot study and data collected during other sampling programs conducted on site, low-flow sampling will be utilized when collecting groundwater quality data.
7				Contingency Plan - If we have to implement the contingency plan, does it take place at the new contingency wells or will additional wells, etc be needed. It was confusing when section 6 said "The final contingency remedy will be designed..."	If a contingency plan must be implemented, it will likely involve utilizing both the contingency wells installed under this program and additional wells installed for the contingency remedy.
Additional Comments from Joel Sanders					
1				Change contingency portion of work plan so that the team decides when to implement contingency.	As discussed during the Charleston DQO meeting, the work plan will be revised to indicate that if the concentration of vinyl chloride (VC) exceeds current regulatory limits plus existing ambient concentrations at the contingency wells, the NIROP partnering team will be consulted to determine if the implementation of a

PARSONS ENGINEERING SCIENCE, INC. RESPONSE TO JOEL SANDER'S COMMENTS					
					contingency remedy is necessary.
2				Make sure Parsons knows how to sample for VC (very low detection limits)	The QAPP will be revised to include the specific detection limits for the approved low detection limit method to be performed by the MPCA/EPA-approved laboratory (EnChem).

PARSONS ENGINEERING SCIENCE, INC.
RESPONSE TO EPA REGION 5 COMMENTS

SITE: NIROP Fridley, Minnesota

DOCUMENT: Final Quality Assurance Project Plan (QAPP) for Field Application to Enhance *In-Situ* Bioremediation of Chlorinated Solvents via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, MN

REVIEWER: L. Finkelberg, U.S. EPA Region 5

RESPONDENT: Prepared by Parsons Engineering Science, Inc.; submitted 08/06/01

Item	Section	Page	Line	Comment	Response
1				General The submitted QAPP is not acceptable: the document should be written based on the EPA Requirements for Quality Assurance Project Plans (Nov. 1999 EPA QA/R-5) and Region 5 Superfund Requirements (June 2000). Please resubmit the document following referenced documents Make sure to include Title and Approval page, provide more site-specific details for site background, current status and contamination (based on previous investigation).	The QAPP will be re-formatted to adhere to the EPA Region 5 guidance entitled: *Region 5 Instructions on the Preparation of a Superfind Division Quality Assurance Project Plan. Based in EPA QA/R-5, Revision 0. June, 2000.*
2	4			The statement " Relative Percent Difference (RPD) calculated between primary and field duplicate samples" is not accurate. Is primary sample not a field sample? Please explain.	The term "field sample" will be substituted for "primary sample" throughout the document.
3	4.1			Section 4.1 listed target RPDs for field duplicate samples as 35% for water and soil samples. We recommend to change target RPD limit for water from 35% to 25 % and keep 35% for soil samples.	Concur, the RPD for field duplicate samples for water will be changed to 25%.

PARSONS ENGINEERING SCIENCE, INC. RESPONSE TO EPA REGION 5 COMMENTS					
Item	Section	Page	Line	Comment	Response
4	5.1			Section 5.1 needs to be revised to be more specific in addressing the accuracy and precision of field measurements.	Field measurements for parameters such as pH, specific conductance, temperature, etc. will be assessed for accuracy in the field. Specifically, field instruments will be assessed for accuracy by the response to a known calibration standard sample. The objective for accuracy of field measurements is to achieve and maintain factory QC specifications for each piece of field equipment.
5	7.1.2			Section 7.1.2 needs to be revised to indicate that trip blank required to accompany the water samples collected for VOA.	Disagree, trip blanks will be required to accompany all VOC samples regardless of matrix per the original QAPP.
6	7.2.8			Section 7.2.8. Please be more site specific and DQO project specific (after the DQO process will be established) in terms of the establishing acceptance criteria for the control limits associated with analytical methods.	Sections have been added to section 2.5 in the latest version of the QAPP to address site specific field measurements control limits. In addition, 4 columns have been added to Table 1.3 to address site analyte specific control limits. Draft site specific DQOs are presented in Attachment 2 of the latest version of the QAPP.
7	7.3.3			Section 7.3.3. References Table 1.2 for Laboratory Reporting Limits for each target compound. In fact, Table 1.2 listed the PQLs for organics (VOA) in water samples only. All other parameters in soil and water matrices are listed as TBD. All Project Required Quantitation Limits must be established prior to the selection of sampling and analytical methods.	Laboratory MDLs and PQLs have been added to Table 1.3.

PARSONS ENGINEERING SCIENCE, INC. RESPONSE TO EPA REGION 5 COMMENTS					
Item	Section	Page	Line	Comment	Response
8	1			The footnote 3/ (Table 1.2) states that PQL will be determined from the subcontracting laboratory. Please address in the QAPP if you are planning to get analytical service from subcontract laboratory. The laboratory name and some credentials of the laboratory experience should be addressed in the QAPP prior the selecting the laboratory.	The current project quantitation limits (PQL) were provided by the subcontract laboratory (Enchem). After the DQOs are finalized, the PQLs will be reviewed to ensure that the PQLs are sufficiently low to satisfy the DQOs
9	7			Table 7.1 needs to be revised to address the preservation for water samples collected for NO3/NO2 analysis. Samples should be cooled to 4C.	Table 7.1 (Table 2.3 in the latest version) has been revised.
10	7			Table 7.2 (page 7-41) needs to be revised to be consistent with the laboratory Quality Control section of SOP. Where is the information about matrix spike /matrix spike duplicate analysis?	Table 7.2 has been revised as recommended.

PARSONS ENGINEERING SCIENCE, INC.
RESPONSE TO EPA REGION 5 COMMENTS

SITE: NIROP Fridley, Minnesota

DOCUMENT: Final Quality Assurance Project Plan (QAPP) for Field Application to Enhance In-Situ Bioremediation of Chlorinated Solvents via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, MN. Revision 3

REVIEWER: L. Finkelberg, U.S. EPA Region 5

RESPONDENT: Prepared by Parsons Engineering Science, Inc.; comments submitted via conference call on 8/21/01 between Venky Venkatesh (CH2M Hill Constructors Inc.) and Luba Finkelberg (EPA Region 5)

Item	Section	Page	Line	Comment	Response
1				General Include Section#, Revision#, Date, Page#, etc. on each page per the QAPP instructions.	This comment will not be addressed as discussed during the above referenced conference call.
2	1.1.2.2			Revise Section 1.1.2.2 to include the following – 1. Reviewing and approving the QAPP 2. Conducting external Performance and System Audits of laboratory and Field Activities 3. Reviewing and evaluating analytical field and laboratory procedures	Section 1.1.2.2 has been revised as recommended.
3	1			Include an Organization Chart in Section 1.1	An Organization Chart was included as recommended.
4	1			Section 1.3, include final DQO notes and revise text accordingly.	The text has been revised as appropriate and the final DQO meeting minutes will be included as an attachment.
5	1			Revise TOC method for soils in Table 1.2	Table 1.2 has been revised as recommended.
6	1			Include vegetable oil media in Table 1.3	Table 1.3 has been revised as recommended.
7	1			Clarify what Control Limit LCS (%) and Control Limit Matrix Spike (%)' means. Ensure that these limits match the SOPs provided by the lab.	Quality control tables provided by Enchem Inc. are included as attachment 3. Table 1.3 has been revised by removing the Control Limit LCS and Control Limit Matrix Spike columns and referring to the tables in attachment 3.

PARSONS ENGINEERING SCIENCE, INC. RESPONSE TO EPA REGION 5 COMMENTS					
8	1			Precision and Accuracy limits for each analysis listed in Table 1.3 should be specified in Section 1.4.3.2 and Section 1.4.3.3.	Parsons ES believes that to specify precision and accuracy limits in the text in Sections 1.4.3.2 and 1.4.3.3 would be redundant. Instead, the reader is referred to Attachment 3 where precision and accuracy standards for each analysis and analyte are tabulated for the readers convenience.
9	2			Provide a sampling numbering system in Section 2.3.1.	A sample numbering system description was added to Section 2.3.2.
10	2			Provide project-specific control limits and percent recoveries for laboratory QC samples are specified in Section 2.5.2.2 (pages 2-8 to 2-12) or ensure they are included in the laboratory SOPs. Also, ensure that they comply with the project DQOs.	Project-specific control limits and percent recoveries for laboratory QC samples are included in the laboratory SOPs provided by Enchem, Inc.
11	2			Update Table 2.3 to include all media (water, soil, and vegetable). Also, specify if vegetable oil will be preserved and the specific holding time.	Table 2.3 has been revised as recommended.
12	2			Ensure that the requirements specified in Table 2.4 is in compliance with the laboratory SOPs.	The laboratory will comply with all requirements stated in Table 2.4.
13	2			Include TOC soil method in Table 2.4.	Table 2.4 has been revised as recommended.
14	2			Table 2-6, The RPD criteria for field duplicate in soil and water in Table 2-6 (page 2-30) should match what is in Section 1.4.3.1.	Table 2.6 has been revised as recommended.

ENCHEM INC. RESPONSE TO EPA REGION 5 COMMENTS
SITE: NIROP Fridley, Minnesota
DOCUMENT: Various Laboratory Analytical SOPS
REVIEWER: L. Finkelberg, U.S. EPA Region 5
RESPONDENT: Prepared by Enchem, Inc.; submitted 08/06/01

I. The deficiencies of the SOP for NO2/NO3 analysis.

Attachment 1 from SOP listed Reporting Limits for NO2/NO3 as 0.2 mg/l. The Method is capable to provide MDL as low as 0.004mg/l and 0.013 mg/l. Please identify the project specific required MDL for those parameters and inform the laboratory.

Attachment 1 listed RPD limits as 4%. The limit is very tight. Is it possible to achieve?

Using Method 300.0 (Ion Chromatography) the EQL is 0.2 mg/L. The current MDL for nitrate is 0.13 mg/L and nitrite is 0.077 mg/L. The control charts have been updated since the information was provided to you. The current RPD limit is 6%. The RPDs are usually within this control limit. If an RPD is outside of the control criteria, the data is qualified by EnChem and described in the case narrative. We are willing to use a project specific RPD control limit that is broader than our in-house generated limit if necessary for this project.

II. The following are comments for the SOPs (3-VOA-5 and G3-VOA-18) from EnChem Inc. for clarifications to improve the usability of these SOPs.

A. The SOP provided from the EnChem is written for the detection of volatile organic compounds by GC/MS in water and methanol extracted soils. How about vegetable oil? Please provide sample preparation and extraction procedure for the analysis of VOC in vegetable oil.

8260 Analysis of Vegetable Oil

Due to the viscosity of oil and the opportunity for contamination, we cannot run the samples neat. If the oil viscosity is thin, we will take an aliquot of the oil volumetrically using a syringe or pipette and dilute in a volumetric flask or syringe containing methanol.

If the oil viscosity is thick, we will take an aliquot of the oil gravimetrically (approximately 1 gram) and transfer it to a vial containing 10mLs of methanol. The extract will be diluted 50x prior to analysis yielding a final dilution factor of 500x. The results will be reported in µg/Kg.

If bi-phasic samples are to arrive from the field, the laboratory must be instructed how to proceed with the analysis. The laboratory needs to know whether to analyze both phases, only the oil phase, or only the water phase.

B. The differences between the laboratory's SOPs and the SW-846 methods are outlined below:

- **Standard Operating Procedure for Volatile Organic Analysis by GC/MS - Methods 5030B/8260B (SOP 3-VOA-5)**

- **Section I-Apparatus and Materials (p. 2 of 15) - SOP identifies the fact that they are using commercial calibration mixes but does not identify them;**

  The SOP has been updated to include this information. Please refer to page 3 and Table 5.

- **Section II-B Preparation of Standards (p. 4 of 15) - SOP does not describe how to prepare the internal standards or surrogate solution, it only provides the final desired concentration.**

  The SOP has been updated to include this information. Please refer to Table 6.

- **Section IV-E Annual Quality Control (p. 10 of 15) - The SOP has no mention of an Initial Demonstration of Proficiency as described in Sec 8.3 of SW-846 Method 8260B.**

  The SOP has been updated to include this information. Please refer to page 2.

- **Section II Preparation of calibration File (p. 5 of 15) - The laboratory included additional QC controls; an Initial Calibration Verification Standard (ICV) sample made at the mid-point calibration concentration using a 2nd Source of Stock Standard solutions is included as part of the initial calibration sequence. Analysis does not proceed until the calibration is acceptable.**

The laboratory routinely performs a second source calibration check standard. The check standard must be within the control criteria established by the laboratory prior to beginning sample analysis. We realize that this self-imposed requirement is in excess of the method requirements. We believe this check standard helps insure that the final results satisfy our client's needs and data quality objectives.

- **Standard Operating Procedure for Analysis of Dissolved Methane, Ethane, Propane, and Ethylene in Ground Water by Static Headspace and Gas Chromatography - Method 5021 (SOP G3-VOA-18)**

This method is an adaptation of a soil/solid waste method; therefore, there is no historical data to reference.

EnChem has performed an IDC Study of the adaptation for the aqueous matrices for which EnChem does analysis. Attached is the IDC study data.

- **The analytes of interest; Methane, Ethane, Ethylene and Propane have not historically been analyzed in soil/solid samples by this method.**

EnChem has written the SOP exclusively for Aqueous samples and is reserved for the detection of the compounds mentioned above.

- **The SOP does not state what is the acceptance criteria for the initial calibration curve, i.e. linearity r 0.995% RSD for response factors 30%, etc.**

The criteria have been added to the next revision of the SOP. Revised SOP attached.

One (1) rounding error was found in the calculated Conversions of Gas Standards:

***x* g/mole X *y* µmole/g X 1000 µg/1000 µmole X 1 mole/22.4 L = gas conc.**

Analyte	g/mole (*x*)	µmole/g (*y*)	Lab's results	Recalculation
Methane	16	100	71	71.42 ~ 71
Ethane	30	105	141	140.625 ~ 141
Ethene	28	101	126	126.25 ~ 126
Propane	44	101	199	198.29 ~ 198 *

This change has been included in the next revision of the SOP. Revised SOP attached.

5. **SOP does not state the criteria for the retention time windows for analyte identification.**

   A retention time study has been performed and attached is the data.

6. **The Laboratory's SOP does not include the addition of internal standards and/or surrogates.**

   RSK 175 does not include information on appropriate IS/SS selection. Due to the molecular nature of the (light weight) an appropriate IS/SS was not found.

7. **As written the SOP is not a stand-alone method that allows an analyst to perform the activities described within and it does not reference any other EnChem SOPs.**

   Please provide a list of items that are considered not in the current SOP that should be included.

III. This is a review of two Standard Operating Procedures for Total Organic Carbon submitted by EnChem, Inc. The SOP's were evaluated using SW 846, method 9060 as a guideline. Included with the SOP's was a table of Flagging Conventions for Data Evaluation and Validation of Inorganic Methods, Table 9.2, which lists the technical requirements of the data user. The SOP's were also reviewed and checked against this table to verify that all conditions were accounted for.

EnChem SOP, WCM-2, Revision No. 9, April 1999:

Table 9.2 gives a requirement for the calibration correlation coefficient curve of the instrument. Table 9.2 also requires the use of multiple points for calibration. The calibration method described in the SOP appears to be a one-point calibration and thus does not produce a calibration correlation coefficient. The lab may need to employ another method of calibration to meet the correlation coefficient requirement. A Dohrman 190 is also used at Region 5, where the analyst analyzes samples and calculates the linear regression offline to demonstrate acceptable linearity. Table 9.2 suggests that all analyses not meeting this requirement are qualified.

The procedure, Step 2, item a, does not give the concentration of the standard used as the calibration standard.

The procedure, Step 4, item c, requires using 3 injections per sample and throwing out the first based on the fact that the first injection is used to prime the combustion tube. If the lab is having problems with reproducibility of results between injections, this may be due to a problem with the plumbing of the instrument and should be investigated. Region 5 CRL uses the same instrument and there has been no need to run samples in the manner described above.

The quality control section gives definitions of all the quality control analyses. The Initial Calibration Verification standard is stated to be from a source different from that used for calibration. The Continuing Calibration Verification should also be from different source than that of the calibration standard; this should be stated.

The Method Blank statement does not give a contingency for corrective action if the blank does not meet the control limits. The Laboratory Control Sample, Matrix Spike, and Matrix Spike Duplicate statements do not tell what the control limits are or what the contingency for corrective action would be if the results were out of control. Corrective action may be required; Table 9.2 requires all results associated with quality control analyses out of control to be qualified.

Since this SOP was sent, we have added another TOC analyzer. It is an Apollo 9000 TOC Analyzer manufactured by Tekmar Dohrmann. We calibrate this instrument using 6 standards and blank for water samples. The correlation coefficient must be greater than 0.995. Aqueous samples are being analyzed using the Apollo 9000. We would like the corresponding SOPs incorporated into the QAPP for use in this project. Copies of WCM-69 and WCM-70 are included. The comments outlined with respect to WCM-2 are addressed in WCM-69 and WCM-70.

EnChem SOP, WCM-9, Revision No. 11, January 2001:

The Apparatus Section, balance description, should include a statement that 0.1 mg resolution and accuracy is required. This is stated in the Dohrman manual.

The SOP has been updated to include this information. Please refer to page 2.

The procedure refers to EnChem SOP, WCM-18 for start up, shut down of the instrument, and set up of Soils/Boat. This SOP was not provided for reference or review.

A copy of WCM-18 is included for reference and review.

Table 9.2 gives a requirement for the calibration correlation coefficient curve of the instrument. Table 9.2 also requires the use of multiple points for calibration. The calibration method described in the SOP appears to be a one-point calibration and thus does not produce a calibration correlation coefficient. The lab may need to employ another method of calibration to meet the correlation coefficient requirement. A Dohrman 190 is also used at Region 5, where the analyst analyzes samples and calculates the linear regression offline to demonstrate acceptable linearity. Table 9.2 suggests that all analyses not meeting this requirement are qualified.

The laboratory is willing to analyze multiple standards to generate a calibration curve and the associated correlation coefficient in order to satisfy this requirement. The linear regression will be calculated off-line to demonstrate acceptable linearity and to insure that the sample concentrations as calculated by the instrument are not qualified.

A calibration curve using a blank, a 500, 1000, 2000, and 3500 ppm standards

The Initial Calibration Verification and the Continuing Calibration Verification standards should be from a different source than that of the calibration standards; this should be stated.

The SOP has been updated to include this information. Please refer to page 3.

No corrective course of action is specified for the occasion when the Initial Calibration Blank is out of control.

The SOP has been updated to include this information. Please refer to page

The Method Blank statement does not give a contingency for corrective action if the blank does not meet the control limits. The Laboratory Control Sample, Matrix Spike, and Matrix Spike Duplicate statements do not tell what the control limits are or what the contingency for corrective action would be if the results were out of control. Corrective action may be required; Table 9.2 requires all results associated with quality control analyses out of control to be qualified.

The SOP has been updated to include this information.

The soil sample preparation may require additional steps. The sample weights need to be recorded before and after treatment with acid. The TOC result then needs to be corrected for the additional weight, which is added. Since the post treatment weight would also be affected by moisture being evaporated, it might be advisable for the samples to be dried prior to treatment. Percent solids would thus need to be determined, and the TOC result would need to be adjusted accordingly. The TOC result needs to be adjusted anyway if the final result is to be reported on a wet weight basis.

Ten grams of soil is treated with acid, dried in an oven at 104°C, and then ground to homogenize the sample. 10 milligram aliquots of soil are weighed out for analysis. The soil should be 100% solid due to the oven drying procedure. The results are reported on a dry weight basis without any correction.

EnChem RESPONSE TO EPA REGION 5 COMMENTS
SITE: NIROP Fridley, Minnesota **DOCUMENT:** Final Quality Assurance Project Plan (QAPP) for Field Application to Enhance In-Situ Bioremediation of Chlorinated Solvents via Vegetable Oil Injection at the Naval Industrial Reserve Ordnance Plant (NIROP), Fridley, MN **REVIEWER:** Submited by Tom Sedlacek for Allison Harvey To Luba Finkelberg, U.S. EPA Region 5 (via e-mail, 08/21/01) **RESPONDENT:** Prepared by EnChem.; submitted 08/29/01

The lab repondence addresses the deficiencies stated in the original review and provides appropriate corrections or supporting information to bring the organic SOP closer to compliance with Region 5 guidelines. The following problems and deficiencies still remain.

A. SOP G3-VOA-18 Stand Operating Procedure for Analysis of Dissolved Methane,Ethane, Propane, and Ethylene in Ground Water.

ORIGINAL REVIEW COMMENT
This method is an adaptation of soil/solid waste method: therefore, there is no historical data to reference.

AFTER LAB COMMENTS AND REVISIONS
The lab provided an IDC study for this method which proves reproducibility at the mid-point of the curve. Table A of the SOP lists MDL and EQL values, how are these numbers generated? The lab did not submit supporting data and as stated above there is no historical data from which to infer a reasonable detection limit.

ENCHEM RESPONSES:
Under the DETECTION LIMITS section of page 1 the following has been added:

> **"The method detection limits were obtained by procedures as outlined in 40CFR Part 136. The spikes were prepared in the same manner as samples. An Estimated Quantitation Limit of 10 ug/L was choosen. This value is above the MDL and within the calibration curve."**

ORIGINAL REVIEW COMMENT
As written the SOP is not a stand-alone method that allows an analyst to perform the activities described within and does not reference other EnChem SOPs.

The lab responded "Please provide a list of items that are considered not in current SOP that should be included."

ADDITIONAL COMMENTS
The lab still has numerous undocumented assumptions in this SOP. It appears that an external calibration method is to be used for the analysis of these gases, but the lab neither states that or includes any reference to size of sample or injection volume. Both of those parameters greatly affect the true sample concentration value when using an external calibration method. The lab needs to state all assumptions and to check all formulae and calculations stated in SOP to ensure that.

ENCHEM RESPONSES:

Under Section 10.1 on page 3 the following was added:

"From these standards an external calibration curve is generated for quantitation."

Under Section 11.3 on page 4 the following was added:

"External Instrument Calibration"

Under Section 11.51 the following was added:

"1.0 mL of the gases from the headspace in the vial are then injected onto the column and moved through the column by a flow of nitrogen."

A copy of the re-reviewed and updated SOP is attached.

B. SOP VOA-5 Volatile Organic Analysis by GC/MS

ORIGINAL REVIEW COMMENT
The SOP provided from EnChem is written for the detection of volatile organic compounds by GC/MS in water and methanol extracted soils. How about vegetable oil? Provide sample preparation and extraction procedure for analysis of VOC in vegetable oil.

AFTER LAB COMMENTS AND REVISIONS.
The lab appears to be using a modification of the medium level soil method for VOC analysis in vegetable oil, but there still are the following problems:

1) It still is not stated in the SOP the procedure to be used.

ENCHEM RESPONSES:
The issue of performing a methanol oil dilution can be added to the SOP. Issues specific to this project can be addressed in an addendum. EnChem is performing research and development on the VOA method in vegetable oil. An Initial Demonstration of Capability study and a Method Detection Limit Study are in

progress. Upon completion, copies of the results and the addendum to the SOP will be provided for review. It is our understanding that several weeks of field work must be completed at the site before any sampling and analysis would occur. The method development would be completed and provided for review within that time period.

2) It is not stated or inmplied how the diluted vegetable oil samples will be analyzed. Will the lab be using the meduim level soil calibration curve or the standard water calibration.

ENCHEM RESPONSES:
The lab will be using a medium level methanol extraction, 4 gram of the sample to 10 mL of methanol, to extract the samples. An aliquot of the methanol extract will be diluted in organic free water using Class A volumetric flasks. The lowest dilution that can be preformed will be a 50x dilution in addition to the 2.5x dilution performed in the extraction. Yielding a final dilution of 125x. The diluted sample will be transferred to a 40 mL VOA bottle for analysis. The lab will be using a standard water calibration curve for quantitation.

3) As stated in its reply the lab will be reporting results for vegetable oil as ug/L and ug/Kg. How is this data to be compared? What are recovery limits for each set of units? What is detection limits for ug/Kg and what is detection limit for ug/L?

ENCHEM RESPONSES:
To simplify reporting issues, the lab will gravimetrically dilute all samples in methanol. The lab would then report the data in ug/Kg. The lab does not have historical data on the vegetable oil matrix. Default limits (e.g. 50 to 150 % recovery) would need to set initially until we have historical data on this matrix. The detection limits have not been determined for this matrix. If the client has proprietary QC data that the firm could share, the lab could evaluate these limits so that our limits are comparable.

4) How is the final sample concentration be be determined for each dilution method.

ENCHEM RESPONSES:
Based upon an extrapolation of Medium level soil calculations, the results for
target analytes will be calculated using the following equation:

Conc. (ug/Kg) = ((Ax) *(AMTis) *(Vt)
(1000)(DF))/((Ais)*(RRF)*(Va)* (W)*(D))

Where:

Ax = area of the characteristic ion of the compound
AMTis = amount of internal standard in ng (250 ng)
D = dry weight factor ((100 - %Moisture)/100)
W = weight of sample extracted in grams
Ais = area of the characteristic ion of the associated internal standard
RRF = analyte's relative response factor from the initial calibration
Vt = Total volume of the extract in mL
Va = Volume of aliquot of extract purged, in mL
DF = Dilution factor

5) In describing the thin viscosity oil scenario, there is no mention of final volume or expected dilution factor. Is there a minimum volume of methanol required to dissolve vegetable oil in water.

ENCHEM RESPONSES:
The oil is not miscible with methanol. The oil will separate into a white layer at the bottom of the vial and the methanol in the top phase is cloudy.

6) Will the lab be running vegetable oil method blanks and which sampling method will the lab be using?

ENCHEM RESPONSES:
The lab will weigh a vegetable oil blank to be used as an extraction blank. The vegetable oil method blanks will be extracted and analyzed using the same procedure as the samples.

7) What is MDL for target compounds in vegetable oil. Lab must submit for all vegetable oil extraction methods.

ENCHEM RESPONSES:
EnChem is performing research and development on the VOA method in vegetable oil. An Initial Demonstration of Capability study and a Method Detection Limit Study are in progress. Upon completion, copies of the results and the addendum to the SOP will be provided for review. It is our understanding that several weeks of field work must be completed at the site before any sampling and analysis would occur. The method development would be completed and provided for review within that time period.

8) What are the acceptable limits for the Initial Demonstration of Proficiency and has the lab demonstrated it can meet those limits?

ENCHEM RESPONSES:
Given the unknown properties of the vegetable oil matrix, EnChem believes that the recovery limits of 80-120% and relative standard deviation (RSD) limit of 20% in the volatile SOP (VOA-5)

are too confining. The limits should be default limits (e.g. 50-150 % recovery and a RPD of 50 %), with the understanding that these limits are subject to change. The Initial Demonstration of Proficiency will be performed at the same time as the MDL study. The acceptance limits will be included in the SOP addendum.

REVISED RESPONSES TO MPCA COMMENTS ON

Superfund and Voluntary Investigation and Cleanup Programs
Sampling Protocol Template For Monitoring Wells at Anoka County Park
Pilot-Scale Study for Enhanced *in-situ* Bioremediation of Chlorinated Solvents via Vegetable Oil Injection

Comments Made by: Paul Estuesta, Minnesota Pollution Control Agency

1. The template represents a procedure for collection samples for evaluation of the bioremediation of chlorinated solvents in the Anoka County Park located across East River Road from NIROP. The MPCA staff requests that the template once completed be considered as the Sampling Standard Operating Procedure (SOP) as identified in the document entitled, "Region 5 Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan, Revision 0," June 2000. To that end I submit the rest of the responses for inclusion the final Sampling SOP.
   Response: Acknowledged. Revised document attached.

2. Section 2.1, Selection of Analytical Parameters: page 5, The MPCA staff requests that this item be Appendix A, not referenced.
   Response: By referencing the Work Plan and the QAPP, the Sampling Template will be a standalone document requiring no changes if the Work Plan or QAPP were to be revised. This will cut down on the cost of document preparation and eliminate any confusion if the document is not updated properly. Additionally, it will be convenient for field personnel to have the latest information in the document (referenced to Work Plan or the QAPP). Note that USEPA Region 5 allows a document to be referenced to avoid redundancy and to minimize the size of the individual document. We hope MPCA understands the logic behind this approach and approves this SOP.

3. Section 2.2, Detection Limits, PQLs: page 5, The MPCA staff requests that this item be in Appendix A, not referenced.
   Response: Same response as in Comment 2.

4. Section 2.8, Order of Sampling: page 7, The MPCA staff requests that this information identified be here and in Table 2.
   Response: Same response as in Comment 2.

5. Section 3.5, Purging and Stabilization: page 9, " *Wells that do not have extremely slow recharge will be purged and sampled as described below. Purging will be conducted in a manner that, to the extent possible, removes all the "old" water in the well so it is replaced by fresh ground water from outside the well installation.*" The MPCA staff requests that the pump be placed approximately two (2) feet above the screened or open portion of the well and be purged at a rate not to exceed the draw down of more than two (2) feet. Also the MPCA staff requests that if pumps are used for purging and sampling, the models and their rate capacities be identified here.
   Revised Response: Based on the teleconference call and e-mail conversations with MPCA on 08/30/01, it was agreed that the pump will be set just below the water table and purged at a rate not to exceed the draw down of two feet. It should be noted that this is not the Navy's preferred method, but this method will be followed to be consistent with October 1999 sampling event. Revised document attached.

6. Section 4.4, Field Blanks and Replicates: page 11, *" Field ambient air blanks will not be required for this project because the project site is a community park and no VOCs will be emitted from an operating facility during sampling. Also, no automobile engines will run during the sampling event."* The MPCA staff requests that if the pumps use a gas-driven generator, the generator be located downwind from the sampling point and the text so indicate this.
   Response: Acknowledged. Revised document attached.

7. Section 4.4, Field Blanks and Replicates: Page 12, Field Replicates, last sentence. The MPCA staff requests that the lab receiving the samples be identified here.
   Response: Same response as in Comment 2.

8. Table 1: Sample Containers, Filling Method, Preservation and Holding Times: page 15. The MPCA staff requests that this table be filled with the information requested, not referenced.
   Response: Same response as in Comment 2.

9. Figure 1: Locations of Sampling Points: page 16, The MPCA requests that the table be filled with the information requested, not referenced.
   Response: Same response as in Comment 2.

10. Appendix A: Selected Analytical Parameters, Methods, Numbers and Reporting Limits: page 17, The MPCA staff requests that the table be filled with the information requested, not referenced.
    Response: Same response as in Comment 2.

Venkatesh, Venky/CLE

From: Sanders, Joel (Efdsouth) [SandersJR@EFDSOUTH.NAVFAC.NAVY.mil]
Sent: August 30, 2001 2:00 PM
To: 'Betcher, John'; Venkatesh, Venky/CLE; 'Henn, Keith'
Cc: 'Richard Kuhlthau'; 'Tom Bloom (Reg. V - EPA)'; Douglas, David; Estuesta, Paul; Sanders, Joel (Efdsouth); 'paulw@baywest.com'
Subject: NIROP well sampling for Vege/Oct 01

CONGRAGULATIONS ALL!!!! - We now have a sampling method that is "acceptable" by the state for the vege oil pilot scale study and the October 01 groundwater sampling. Sampling just below the top of the water column is not the Navy's first choice (pump at middle of screen), but we will use this method for the vege pilot scale and Oct 01 sampling events because it is represenative of the Oct 99 sampling.

The 2002 sampling method for the new groundwater sampling DQOs is still an issue that will be discussed at the September 01 DQO meeting.

Venky - Please print this is email until you get final approval on the Sampling Protocol.

In summary, the pumps will be located near the TOP of the water column vs 2 FEET ABOVE the well screen. See below for further details.

Joel

-----Original Message-----
From: Betcher, John [mailto:john.betcher@pca.state.mn.us]
Sent: Thursday, August 30, 2001 12:53 PM
To: 'Venky Venkatesh'; 'Henn, Keith'
Cc: Sanders, Joel (Efdsouth); 'Richard Kuhlthau'; 'Tom Bloom (Reg. V - EPA)'; Douglas, David; Estuesta, Paul
Subject: FW: past procedures NIROP well sampling

Venky & Keith - Dave, Paul and I have reviewed the purge and sample method outlined by Tim Ruda in Joel's mail message. This method looks acceptable to us and is apparently consistent with the method used in the 1998-99 sampling events. The method should ensure the removal of stagnant water in the casing (by removing 3-5 well vols.) and gives us confidence that we are sampling a representative groundwater sample from the aquifer without undue influence from where the pump might be set within the screened interval. The higher purge rates will reduce the purge times. In addition, I know that some of the deep wells were purged for the 1999 AMR at a higher rate than 6 gpm (I recall up to 11 gpm) which is OK, as long as 1-2 feet of drawdown is not exceeded. This will not probably be needed for the Veg Oil wells as they are fairly shallow.

It is possible that for some shallow wells that the water level will be within the screen. If this is the case, the same procedure for purging and sampling can be used although in some cases the pump will have to be within the screened interval due to necessity.

If this resolves the issue that came up in this mornings call we probably don't need a conference call. Venky, if you have any questions please give me a call.
I hope this resolves the purge and sample SOP for everyone. For TtNUS this is the SOP that should be carried forward into the OU1 QAPP although the immediate issue at hand is the Veg Oil work plan and QAPP. Thanks. JTB
> ----------

> From: Sanders, Joel
(Efdsouth)[SMTP:SandersJR@EFDSOUTH.NAVFAC.NAVY.mil]
> Sent: Thursday, August 30, 2001 9:44 AM
> To: 'david.douglas@pca.state.mn.us'; John Betcher (E-mail)
> Subject: FW: past procedures NIROP well sampling
>
> Dave - Below is the info Tim Ruda provided us on past sampling on August
3,
> 2001.
>
> Joel
>
> -----Original Message-----
> From: Sanders, Joel (Efdsouth)
> Sent: Friday, August 03, 2001 3:17 PM
> To: 'vvenkate@ch2m.com'; 'sladicm@ttnus.com'; 'hennk@ttnus.com'
> Cc: Sanders, Joel (Efdsouth)
> Subject: FW: past procedures NIROP well sampling
>
>
> FYI - Keep this in mind when we receive MPCA comments. Thanks-Joel
>
> -----Original Message-----
> From: TIM_RUDA@udlp.com [mailto:TIM_RUDA@udlp.com]
> Sent: Friday, August 03, 2001 3:08 PM
> To: Sanders, Joel (Efdsouth)
> Subject: past procedures NIROP well sampling
>
>
> The details of the sampling in 98-99 were:
>
> Set pump at top of the water column (1 foot below water column). The
flow
> rate
> varied depending on well yield. Basically we set the pump at the top of
> the
> water column yet set the pump so we would se a slight drawdown (less than
1
> foot). and then maintained that flowrate at the that water level. In
the
> deep
> wells 6 gpm, intermediate well 2-3 gpm, shallow 1-.5 gpm. all wells were
> sampled with pump turned down to around 500 ml minute. 3-5 well volumes
> were
> pulled out in compliance with stabilization parameters. We do have
sampling
> logs
> to tell us what flowrate at each well. The USGS (Don V.) may have
additional
> field notes of the wells the last time they were sampled in 99. let me
> know if
> you need something more detailed. The field logs should be contained in
99
> AMR.
>
>

MEMORANDUM **SRT-4J**

DATE : 09/17/2001

FROM: L. Finkelberg, Chemist ,
Field Services Section

TO: Tom Bloom, RPM

I am providing conditional approval of the QAPP for Field Application to enhance in-situ bioremediation of chlorinated solvents via vegetable oil injection at the Naval Industrial Reserve Ordinance Plant (NIROP, Fridley, MN which was received by FSS (Log-in No.2746).
The final approval will be issued after the commercial laboratory provide the laboratory procedure for preparation and analysis of vegetable oil samples.