

QUALITY ASSURANCE PROJECT PLAN

for the

**COOL-OX™
TECHNOLOGY DEMONSTRATION
AT
ASHLAND MGP
LAKEFRONT SITE**

ASHLAND, WISCONSIN

DEMONSTRATION PROGRAM

October 2006

**U.S. Environmental Protection Agency
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QUALITY ASSURANCE PROJECT PLAN DISTRIBUTION LIST

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LIST OF ACRONYMS, ABBREVIATIONS, AND SYMBOLS

Fg/kg	Micrograms per kilogram
Fg/L	Micrograms per liter
ASTM	American Society for Testing and Materials
bgs	Below ground surface
°C	Degrees Celsius
CCV	Continuing calibration verification
cm/sec	Centimeters per second
COC	Contaminants of concern
DNAPL	Dense non-aqueous phase liquid
EPA	U. S. Environmental Protection Agency
GC/MS	Gas Chromatography/Mass Spectrometry
HSP	Health and Safety Plan
ICV	Initial calibration verification
IDW	Investigation derived waste
IS	Internal standard
ITER	Innovative Technology Evaluation Report
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
mg	Milligram
mg/kg	Milligram/kilogram
mg/L	Milligram/liter
mL	Milliliter
mL/min	Milliliter per minute
MGP	Manufactured gas plant
MS/MSD	Matrix spike and matrix spike duplicate
NAPL	Nonaqueous phase liquid
NIST	National Institute of Standard and Technology
ORP	Oxidation-reduction potential
PAH	Polynuclear aromatic hydrocarbon
PID	Photoionization detector
PPE	Personal protective equipment
ppm	Parts per million
PRRL	Project required reporting limit
QA	Quality assurance
QAPP	Quality assurance project plan
QC	Quality control
RPD	Relative percent difference
SITE	Superfund Innovative Technology Evaluation
SVOC	Semivolatile organic compound
TSA	Technical System Audit
USCS	Unified Soil Classification System
VOC	Volatile organic compound

SITE QUALITY ASSURANCE PROJECT PLAN

The U.S. Environmental Protection Agency (EPA) established the Superfund Innovative Technology Evaluation (SITE) Program to accelerate development, evaluation, and use of innovative technologies to remediate hazardous waste sites. The evaluation portion of the SITE Program focuses on technologies in the pilot- or full-scale stages of development. The evaluations are intended to provide performance data of known quality, and sampling and analytical procedures are critical. Approved quality assurance and quality control procedures must be stringently applied throughout the evaluation.

Tetra Tech EM Inc. (Tetra Tech) prepared this quality assurance project plan (QAPP) to follow the guidelines in the EPA National Risk Management Research Laboratory (NRMRL) requirements document entitled *QAPP Requirements for Applied Research Projects* (EPA 1998) as well as *Preparation Aids for the Development of Category II Quality Assurance Project Plans*, EPA/600/8-91/004 (EPA 1991). This QAPP describes how the SITE project team will collect and analyze samples to evaluate the Cool-Ox™ technology as deployed by the vendor DeepEarth Technologies, Inc. Tetra Tech prepared this QAPP for EPA under Contract No. 68-W-02-034, Work Assignment 65.

1.0 PROJECT DESCRIPTION

This quality assurance project plan (QAPP) has been prepared to provide guidance for evaluating the application of the Controlled In-Situ Chemical Oxidation (Cool-Ox™) process at the Northern State Power, a subsidiary of Xcel Energy, Lakefront Site (Lakefront Site).

The Cool-Ox™ technology is a chemical that reportedly destroys organic contamination by means of chemical oxidation and accelerates biodegradation. The U.S. Environmental Protection Agency (EPA) Superfund Innovative Technology Evaluation (SITE) Program will conduct an evaluation of the Cool-Ox™ technology.

This section provides site background information and summarizes previous investigations; describes the technology; provides the project objectives; and provides the project schedule. Section 2.0 describes the project organization and participants. Section 3.0 provides details of EPA's evaluation design and statistical approach to evaluate the technology. Section 4.0 describes EPA's field sampling program. The remaining sections describe analytical methods and quality assurance (QA) and quality control (QC) objectives and procedures. Tables and figures are provided at the end of the QAPP.

1.1 SITE SETTING AND HISTORY

The Lakefront Site is a former manufactured gas plant (MGP) located at 301 Lake Shore Drive East in Ashland, Wisconsin. The facility lies approximately 1,000 feet southeast of the shore of Chequamegon Bay of Lake Superior (Figure 1-1). The site soils are contaminated with coal tar and dense nonaqueous-phase liquids (DNAPL) has been observed in site monitoring wells.

The site has been the location of industrial activities over the past 150 years. The site currently consists of a filled area which forms a flat terrace adjacent to the Chequamegon Bay. The surface elevation of the terrace is approximately 600 feet (ft) mean sea level (MSL) adjacent to the bay and rises to approximately 610 ft MSL at the base of the bluff overlooking the bay. The current layout of the site is presented on Figure 1-2.

From 1885 to 1947 an MGP plant operated on the upper bluff at the present location of Northern State Power's garage. Coal tars were produced during this time as a normal co-product. It is estimated that approximately 590,000 gallons of coal tar were produced at the site. Records also indicate that the coal tar product was also used on site for energy recovery (URS 2005).

During the early history of the MGP, a ravine was present and trended north from Lake Shore Drive to the bluff face. By 1909, the ravine was filled to the surrounding grade. Previous investigations indicate that the ravine Fill consists of clays, cinders, and rubble.

1.2 SITE GEOLOGY AND HYDROGEOLOGY

Unconsolidated deposits at the site consist of, in descending order, Fill, the Miller Creek Formation, and the Copper Falls Formation. Fill materials include wood slabs, sawdust, concrete, bricks, bottles, steel, wire, cinders, and earthen fill. The Fill is more than 20 ft thick in the area of the former ravine. Beyond the flanks of the ravine the Fill is generally less than 3 ft thick. The permeability of the Fill is highly variable and ranges from 0.1 to 5×10^{-5} centimeters per second (cm/sec) (URS 2005).

The Miller Creek is a fine-grained clayey silt to silty clay till. The unit ranges in thickness from 7 to 40 ft thick and exhibits a permeability on the order of 3×10^{-6} to 4×10^{-8} cm/sec. The unit is considered an aquitard that separates the overlying Fill from the underlying Copper Falls Formation (URS 2005).

The Copper Falls Formation underlies the Miller Creek Formation. The Copper Falls Formation consists of clean sands with occasional gravel. The hydraulic conductivity of the unit ranges from 5.9×10^{-4} to 9.6×10^{-4} cm/sec. The unit is more than 150 ft thick in the vicinity of the site.

The water table is generally found within the Fill in the ravine or the Miller Creek Formation where the Fill is absent. The horizontal gradient is to the north toward Chequamegon Bay. The vertical hydraulic gradient between the water table and the underlying Copper Falls aquifer is generally downward in the vicinity of monitoring well MW-15. The gradient changes from downward to upward near the bluff face. Near the bay, groundwater elevations in the Copper Falls aquifer are approximately 17 ft above ground surface. A hydrogeological cross section depicting the vertical distribution of groundwater elevations is presented on Figure 1-3.

1.3 SITE CONTAMINATION

Investigations at the MGP site have identified subsurface contamination consisting of dissolved-phase contaminants in groundwater and free-phase DNAPL. One to 2 ft of coal tar is reported to be present at the base of the ravine (URS 2005). The lateral extent of DNAPL contamination in the vicinity of the ravine is presented on Figure 1-4.

According to NewFields Inc. (NewFields), the consultant for the site owner, DNAPL in the vicinity of monitoring well MW-15 has migrated downward through the aquitard and is now present in the Copper Falls aquifer. As a consequence of the upward hydraulic gradient further north and the near neutral buoyancy of the DNAPL, the contamination has remained in the upper portions of the Copper Falls aquifer and is concentrated in the vicinity of monitoring well MW-13. The vertical distribution of contamination is presented on the hydraulic cross section presented on Figure 1-3.

A database of contaminants that have been detected in soil and groundwater at the site was provided to the SITE Program by NewFields. Those contaminants detected in soil in groundwater from the ravine are summarized on Tables 1-1 and 1-2, respectively. Contaminants of concern (COCs) for this demonstration are provided on Table 1-3.

1.4 INTERIM COAL-TAR RECOVERY SYSTEM

The interim coal-tar recovery system consists of three extraction wells (EW-1, EW-2, and EW-3), an oil-water separator, DNAPL storage tank, and wastewater treatment plant is currently operating at the site. The system typically operates at less than 1 gallon per minute. Since the system was installed in 2001, approximately 8,000 gallons of DNAPL and 1,400,000 gallons of water has been recovered.

The extraction wells remove an emulsified mixture of groundwater and DNAPL from the Copper Falls aquifer. The mixture is transferred to an oil-water separating tank with an air bubbler to separate the DNAPL from the water. Contractors for Northern States Power service the recovery system on a weekly basis. Service of the system includes pumping DNAPL from the bottom of the oil-water separator to the 1,000-gallon NAPL holding tank. The thickness of DNAPL in the tank is measured after each pumping event.

The level of DNAPL in the tank is measured by lowering a clean measuring tape through the access hole on the top of the tank to the bottom of the tank. The tape measure is withdrawn and the thickness of the DNAPL interval is determined by the length of product coating the tape. The value is recorded to the nearest tenth of an inch. The previous week's measurement is subtracted from the current week's measurement to determine the increase in DNAPL height during the week. The increase in DNAPL height is multiplied by 16.3 to determine the gallons of DNAPL recovered during the week.

According to the contractors, the holding tank contains a thin skim of material floating on top of water. DNAPL is present below the water in the bottom of the tank. The DNAPL measurement procedure is complicated by the floating skim which tends to coat the tape as it is withdrawn and makes differentiating the DNAPL from water difficult. Thus, there is some subjectivity to the measurements.

Measurements of the volume of groundwater and DNAPL recovered from February 2001 to August 2006 were provided to the SITE Program by NewFields. The data indicate that week to week measurements of DNAPL recovery commonly vary by a factor of four. The average recovery rate considered on an annual basis has progressively declined from approximately 12.4 to 1.7 gallons per day (Northern States Power Company 2006).

1.5 COOL-OX™ TECHNOLOGY AND PROCESS

Cool-Ox™ is a registered trademark of DeepEarth Technologies, Inc. (DTI). Cool-Ox™ is an *in situ* remediation technology that combines controlled chemical oxidation with accelerated biodegradation subsequent to oxidation phase. According to DTI, Cool-Ox™ has three major advantages over competing technologies. They are as follows:

- Contaminants are converted to surfactants
- The pH is basic (approximately 8)
- The process does not generate heat

The process is based on the use of solid peroxygens in aqueous suspension. The peroxygens are injected into the contaminated zone and gradually dissolve and react with water to generate hydrogen peroxide.

DTI believes the slow dissolution rate causes the peroxygens to stay in the contaminated media for an extended period of time, thereby increasing the radius of influence and the probability of contacting contaminants. Additionally, the ongoing generation of molecular oxygen enhances the proliferation of aerobic microorganisms.

The Cool-Ox™ formulations include compounds that activate catalytic metals intrinsic in the soil matrix being treated. This eliminates the need to artificially introduce iron salts. According to DTI, a broad family of troublesome recalcitrant compounds, such as creosotes, pentachlorophenol, polychlorinated biphenyls, polynuclear aromatic hydrocarbons (PAH), fuel hydrocarbons, dioxins, and a host of herbicides and pesticides are treatable with the Cool-Ox™ process.

1.6 SITE-SPECIFIC CONFIGURATION

The areas around MW-15 and MW-13 have been selected for implementation of the Cool-Ox™ process. Figure 1-2 shows the locations of the treatment areas, and Figure 1-3 shows the vertical intervals for Cool-Ox™ injection. For purposes of deployment at the site, DTI and the site owner have stated the following objectives:

1. Determine the optimum injection techniques necessary to achieve the maximum reduction of contaminant mass.
2. Determine the ability of the technology to reduce contaminant mass, including stimulation of intrinsic hydrocarbon degraders.
3. Assess the affect of the technology on the artesian character of groundwater in the Copper Falls aquifer.
4. Determine the effect of the technology on the DNAPL source, including the probability that the treatment will increase the efficiency of the extraction wells near the MW-13 well nest.

The following subsections discuss the rationales for the selection of the MW-15 and MW-13 areas and the general SITE demonstration approach.

1.6.1 MW-15 Area

The MW-15 area was selected for treatment because of the proximity of the former gas holders and because DNAPL has been observed in monitoring well MW-15. This area is on the upgradient end of the

primary source area. Thus, the potential for contaminant contributions from upgradient sources is minimized.

Approximately 24 direct push borings will be installed on an eight-ft grid (Figure 1-5). The presence of subsurface utilities or other obstructions may require individual injection points to be slightly adjusted. The injection probe will be driven to a depth of 40 ft below ground surface (bgs) and withdrawn to 35 ft bgs. Cool-Ox™ reagent will be injected from 40 to 35 ft, 35 to 30 ft bgs, and 30 to 25 ft bgs. The 25-ft-bgs depth corresponds to the bottom of the Miller Creek aquitard. Grout will be injected from 25 to 15 ft bgs. Cool-Ox™ reagent will be injected above the aquitard from 15 to 10 and 10 to 5 ft bgs. The remaining portion of the borehole will be grouted to ground surface.

DTI will initially install the borings in a predetermined sequence. Injection of the reagent will be halted periodically to monitor the borings for foaming. DTI considers the foaming to be a diagnostic tool that indicates contamination. DTI may adjust the deployment methods based on its assessment of the foam. DTI estimates that it will take approximately two weeks to complete treatment of the MW-15 area.

1.6.2 MW-13 Area

The MW-13 area will be treated after completion of the MW-15 area. The MW-13 area was selected for treatment because it is located in the central portion of the DNAPL source area in the Copper Falls aquifer. Three of the four extraction wells at the site are located in or near the MW-13 area and are actively pumping from the Copper Falls aquifer. As discussed in Section 1.4, approximately 8,000 gallons of DNAPL has been removed from this area.

DTI will install 27 injection points will be on a 9.5-ft grid (Figure 1-6). As in the MW-15 area, boring locations may be adjusted where subsurface utilities are present. The borings will extend to approximately 80 ft bgs. The Cool-Ox™ reagent will be injected from the base of the boring to the bottom of the Miller Creek aquitard, with most of the reagent injected below the DNAPL. The borehole will be grouted from the base of the Miller Creek aquitard to within 5 ft of ground surface.

The rationale for injecting the reagent below is that the upward hydraulic gradient will transport the reagent into the DNAPL source volume and toward the extraction wells. DTI believes this will mobilize

the DNAPL by generating surfactants and will enhance recovery of the existing extraction system.

1.7 PROJECT OBJECTIVES

The technology evaluation has both primary and secondary objectives. Primary objectives are considered critical for the technology evaluation, while secondary objectives provide additional information that is useful, but not critical. Each objective is described in the following paragraphs. Details of the experimental approach are provided in Section 3.0. Sampling procedures are described in Section 4.0 and analytical procedures are described in Section 5.0.

1.7.1 SITE Primary Objective

The following primary objective (P1) of the SITE demonstration is considered critical to the success of the evaluation of the Cool-Ox™ process at the Lakefront Site.

P1 Determine if injection of the Cool-Ox™ Technology will measurably reduce the concentration of individual COCs in soil in the MW-15 area.

The SITE Program will evaluate this objective by collecting 45 soil samples prior to injection of the reagent. Post-treatment sampling will consist of two sampling events conducted at 30 and 90 days after treatment. Forty-five soils samples will be at each soil sampling event. Post-treatment samples will be collected as close as practicable as the pre-treatment samples. The samples will be analyzed for volatile organic compounds (VOCs) and semivolatile organic compounds (SVOCs). A statistical analysis will be conducted on the pre-treatment and 90-day-post-treatment data sets to evaluate the change in concentration of COCs.

Three soil samples will be collected from each of the 15 borings shown on Figure 1-7. Pre-and post-treatment borings will be installed as close as practicable. Pre- and post-treatment soil samples will be collected from the same depths. Soil samples will be collected from above the aquitard. The SITE Program's boring will not penetrate through the aquitard.

The statistical approach is described in Section 3.0, the field methods are described in Section 4.0, and the laboratory methods are described in Section 5.0.

1.7.2 SITE Secondary Objectives

The secondary objectives of the SITE demonstration are discussed below.

S1 Evaluate the ability of the suspended chemical oxidants within the Cool-Ox™ reagent to adequately penetrate the subsurface in the MW-15 area.

Bromide is not anticipated to be present in the groundwater at the site. The SITE Program will collect baseline groundwater samples from MW-15 and MW-15A to establish the presence or absence of bromide prior to Cool-Ox™ treatment of the MW-15 area. Assuming bromide is not detected in the pre-treatment groundwater samples, the SITE Program will install 13 post-treatment borings equidistant from the Cool-Ox™ injection borings, and vertical profiling of the groundwater above the aquitard will be conducted. Groundwater samples will be collected from the depths that corresponds to the center of each Cool-Ox™ injection interval above the aquitard. The groundwater samples will be analyzed for bromide and hydrogen peroxide. The vertical profile samples will be collected approximately 30 days after the Cool-Ox™ injection is complete in the MW-15 area.

S2 Evaluate vendor claims that the Cool-Ox™ process will accelerate biodegradation.

Groundwater samples will be collected from monitoring wells MW-15 and MW-15A on three occasions prior to Cool-Ox™ treatment of the MW-15 area. The sampling events will be conducted approximately 7 days apart. Two post-treatment sampling events will occur at approximately 30 and 90 days after treatment. The samples will be analyzed for heterotrophic plate counts, PAH degraders, dissolved oxygen, nitrate (NO₃), sulfate (SO₄), ferrous iron (Fe²⁺), dissolved manganese (Mn²⁺), total inorganic carbon, total organic carbon (TOC), biochemical oxygen demand (BOD), and chemical oxygen demand (COD).

Soil samples will be collected once prior to Cool-Ox™ treatment and twice after treatment to fulfill the P1 objective. The SITE Program will collect and analyze 22 soil samples from each event for heterotrophic plate counts and PAH degraders.

S3 Evaluate vendor claims that the Cool-Ox™ technology does not require the addition of iron or other catalytic metals.

Forty-five soil samples will be collected from the 15 pre-treatment borings shown on Figure 1-7. The samples will be analyzed for metals. Baseline groundwater samples will be collected from MW-15 and MW-15A. The groundwater samples will be analyzed for metals on one occasion. The approach for evaluation of the data is described in Section 3.0. The analytical methods are discussed in Section 5.0.

S4 Monitor the concentration of dissolved phase VOCs and PAHs in the MW-15 area.

Groundwater samples collected for the S2 objective will also be analyzed for VOCs and SVOCs on three occasions prior to treatment and at 30 and 90 days after treatment. The approach for evaluation of the data is described in Section 3.0. The analytical methods are discussed in Section 5.0.

S5 Evaluate the vendor claim that surface foam emanations are an indicator of contamination and are helpful in increasing the effectiveness of the Cool-Ox™.

Seven of the borings that will be installed for the P1 objective will be collocated with Cool-Ox™ injection points. The soil samples that are collected will be visually described and evidence of contamination such as a sheen or chemical odors will be documented in the field book. Deployment of the Cool-Ox™ process in the MW-15 area will be observed by the SITE Program to document how the deployment is modified as a consequence of the formation of the foam mushrooms. The approach for evaluation of the data is described in Section 3.0. The analytical methods are discussed in Section 5.0.

Up to five soil borings may be installed after treatment to evaluate locations where foaming was observed to test the effectiveness of the process or at locations where the foam was not observed but contamination is suspected to be present and to be ineffectively treated. This decision will be made based on observations made in the field and the analytical results of soil and groundwater samples collected in the MW-15 area.

S6 Evaluate the temperature and pH changes in the subsurface in the MW-15 and MW-13 areas due to the Cool-Ox™ treatment.

Temperature and pH measurements will be taken at monitoring wells MW-15 and MW-15A each day for 7 days after treatment is complete in the MW-15 area. Likewise, temperature and pH measurements will be collected from MW-13A after completion of treatment in the MW-13 area. Attempts will be made to collect measurements from MW-13B as well. However, MW-13B is reported to be damaged, and

previous attempts to collect groundwater quality data were unsuccessful.

S7 Evaluate the change in DNAPL recovery in the MW-13 area as a consequence of the Cool-Ox™ injection.

The SITE Program will conduct measurements of DNAPL recovery on three occasions seven days apart prior to injection in the MW-13 area. Measurement points include the existing 1,000 gallon holding tank, MW-13A, EW-1, EW-2, and EW-3. Post-treatment monitoring will include measurement events at 30 and 90 days after treatment of the MW-13 area.

Post-treatment monitoring will consist of collection of DNAPL recovery measurements at the existing treatment system on a weekly basis for 90 days after injection of the reagent in the MW-13 area. These data will be collected by the site owner's contractor, NewFields, as part of its routine monitoring of the extraction system. NewFields will collect the data in accordance with the procedures specified in Section 4.0 of this QAPP.

S8 Evaluate vendor claims that Cool-Ox™ injection will generate *in situ* surfactants and will mobilize and solubilize contaminants in the MW-13 area.

DNAPL samples will be collected from the 1,000 gallon holding tank, MW-13, EW-1, EW-2, and EW-3 on three occasions prior to treatment of the MW-13 area and 30 and 60 days after treatment. The samples will be analyzed for surface tension, density, and viscosity. One pre-treatment sample and two post-treatment samples collected from the 1,000 gallons holding tank will be submitted for analysis of VOCs, SVOCs, and total petroleum hydrocarbon -extractables (TPH-e). Additional samples may be submitted for chemical analysis if observations suggest that the character of the DNAPL is changing significantly. This decision will be made after consultation with the project chemist and the EPA Work Assignment manager (WAM).

The implementation schedule for the Cool-Ox™ Demonstration is provided on Table 1-4.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The success of this SITE evaluation project depends on a cooperative effort involving government and private parties. This section identifies these parties and describes their roles. Project organization is presented on Figure 2-1.

2.1 EPA WORK ASSIGNMENT MANAGERS

Paul McCauley and Jim Cummings will share responsibilities as the EPA WAMs. The WAM has overall responsibility for the SITE evaluation project. The responsibilities of the EPA WAM for the evaluation include the following:

- Provide technical direction to the EPA technical support contractor, Tetra Tech EM Inc. (Tetra Tech), during all phases of the project
- Plan and coordinate meetings and communications among the various parties involved in this evaluation
- Review and comment on the proposed DTI procedures
- Review and audit field activities
- Ensure that technology evaluation requirements are met
- Review and approve the QAPP and all final reports

2.2 EPA QA MANAGER

EPA places an emphasis on QA and QC for all SITE projects. As a prime contractor to EPA, Tetra Tech is responsive to the QA requirements of EPA and has instituted a parallel organization designed to achieve established EPA goals.

The EPA QA Manager, Scott Jacobs, is responsible for overseeing and reviewing project QA activities in support of the EPA WAM. He is also responsible for communicating EPA QA policy and guidance to Tetra Tech through the Tetra Tech QA Manager, Dr. Greg Swanson. The responsibilities of the EPA QA Manager for the evaluation include the following:

- Review and comment on the QAPP and all final reports

- Set policy and provide guidance for the conduct of field and laboratory audits, and provide oversight for all field and laboratory audits
- Provide general QA guidance and consultation on an as-needed basis throughout this evaluation

2.3 TETRA TECH SITE PROJECT MANAGER

EPA has contracted Tetra Tech to provide comprehensive technical support for this evaluation project. The Tetra Tech project manager, Younus Burhan, is responsible for all tasks performed by Tetra Tech and for direct communication with evaluation participants. John Vanover will assist Mr. Burhan with technical issues as they arise. Mr. Burhan is also responsible for ensuring that all sampling, analytical, and QA/QC requirements are met for the project. Mr. Burhan will prepare technical documents and coordinate technical communications with EPA, DTI, and the site owner or NewFields. In addition, Mr. Burhan will review sampling and analytical data obtained during the evaluation and will be responsible for preparing the final report. Mr. Burhan's specific responsibilities include the following:

- Communicate with and receive technical direction from the EPA WAMs
- Develop the QAPP, innovative technology evaluation report (ITER), and other project deliverables
- Manage staff
- Provide required planning, cost, and schedule control
- Maintain project file and written records documentation

2.4 TETRA TECH SITE QA MANAGER

Dr. Greg Swanson is Tetra Tech's contract QA manager. Dr. Swanson will coordinate QC technical operations among project staff. Dr. Swanson's specific responsibilities include the following:

- Provide assistance and guidance in developing and revising the QAPP
- Provide guidance and coordination to rapidly resolve QC problems
- Review the quality of all project documentation, including data packages and reports

Dr. Swanson is specifically responsible for reviewing and ensuring the quality of all project deliverables.

2.5 THE TECHNOLOGY OPERATOR

DTI is responsible for implementation of the Cool-Ox™ process. DTI will be responsible for supervising the technology operations in accordance with the requirements outlined in this QAPP. DTI will coordinate evaluation activities with EPA to ensure that all requirements are met. DTI will be responsible for the following:

- Injection of reagent into the MW-15 area
- Injection of the reagent into the MW-13 area
- Review the QAPP and all final reports
- Provide technical data, and cost data, as needed by EPA

2.6 LABORATORY ANALYSIS

Tetra Tech will use the following laboratories for this demonstration:

- \$ Columbia Analytical Services, Inc.
- \$ Microbe Inotech Labs
- \$ Torkelson Geochemistry Inc.

The samples will be analyzed in accordance with the procedures, methods, and detection limits specified in Sections 5 and 6. Assigned project leads from each laboratory will be responsible for the following where appropriate or required:

- Provide the sample containers, trip blanks, labels, shipping documents, custody seals, chain-of-custody forms, and shipping containers specified in this QAPP and other supplies as requested by the field sampling team
- Analyze all samples shipped to the laboratory for the parameters specified in the QAPP
- Implement appropriate QA activities, including validation of all analytical work, to ensure compliance with this QAPP
- Provide a validatable data package, including all method-specified QA and QC samples, raw data, calibration data, sample preparation data, standards preparation data, and narrative report

The assigned QA representative from the laboratory will be responsible for reviewing laboratory

procedures, assisting with external laboratory audits, ensuring that laboratory chain-of-custody procedures are followed, ensuring that QAPP protocols are adhered to and that any deviations are documented, conducting internal audits of the laboratory, and assisting in QA review of data to ensure that project measurement objectives are met. Soil, groundwater, and DNAPL samples will be analyzed for VOCs, SVOCs, metals, hydrogen peroxide, and bromide. The laboratories that will be conducting the analyses are briefly discussed in the subsections that follow.

2.6.1 Columbia Analytical Services, Inc.

Columbia Analytical Services, Inc. (Columbia) located in Kelso, Washington, will serve as the laboratory performing the chemical analysis of soil, groundwater, and DNAPL during the evaluation study. The Columbia laboratory project manager, Mr. Howard Boorse, will be responsible for overall supervision of laboratory analyses and reporting to the Tetra Tech project manager.

2.6.2 Microbe Inotech Laboratories

Microbe Inotech Labs (MIL) will perform biological analyses of soil and groundwater samples collected during the demonstration of Cool-Ox™. Mr. Bruce Hemming will serve as overall project manager for MIL. Analyses will be performed in St. Louis, Missouri.

2.6.3 Torkelson Geochemistry Inc

Torkelson Geochemistry Inc. will perform the analyses on the physical characteristics of DNAPL for this demonstration. Mr. Bruce Torkelson will serve as overall project manager for Torkelson. Analyses will be performed in Tulsa, Oklahoma.

2.7 PERSONNEL LOCATIONS

The locations and telephone numbers of the Cool-Ox™ technology evaluation participants are as follows:

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3.0 SITE EXPERIMENTAL APPROACH

The experimental approach includes the various tasks that will be conducted to collect the data needed to accomplish the overall project objectives. This section describes the data that will be collected before and after injection of the reagent. The number of samples and analytical methods for each objective are provided on Table 3-1. The rationale for the number of samples is provided in the sections that follow.

3.1 PRIMARY OBJECTIVE P1

The P1 objective is considered critical to the success of the evaluation of the Cool-Ox™ technology at the Lakefront Site. The objective is to determine the ability of Cool-Ox™ process to reduce concentrations of COCs in the soil in the MW-15 treatment area. Enough samples must be collected to provide a reasonable expectation that meaningful conclusions can be reached concerning the project objective. At the same time, cost constraints limit the number of samples that can be collected and analyzed. To that end, the anticipated statistical strength of the proposed numbers of samples was considered. The statistical analysis is described in the following subsection.

3.1.1 Statistical Evaluation of Existing Data

As summarized in Section 1.0, soil and groundwater analytical data collected during previous investigations were provided to EPA by NewFields. The investigations were conducted for the purpose of identifying the extent, and to some degree, the magnitude of contamination; consequently, the samples were collected in uncontaminated, marginally-contaminated, and heavily contaminated areas. A subgroup of soil samples from the ravine Fill in the vicinity of MW-15 was used for statistical analysis to estimate the number samples required to support objective P1. This data set consists of nine samples from six borings, and is presented Table 3-2. The data set includes the seven COCs.

A broad range of soil concentrations is observed in the data. These concentrations illustrate the extreme heterogeneity of contamination in the Fill. Ranging from the low parts per billion (ppb) to above 1,000,000 ppb for target PAHs, and above 100,000 ppb for target VOCs. Normality testing by the Shapiro Wilk test indicated that the most of the data sets were lognormally distributed. Thus the log transformed data sets became the basis for calculation of sample requirements.

Further calculations to estimate sampling requirements focused on benzo(a)pyrene (BaP) because of its status as a carcinogenic, and because its mean concentration and variance were in the middle of the ranges observed for PAHs and VOCs near MW-15. The objective of the calculation was to estimate the number of soil samples that would be sufficient to discern a measurable difference in the pre- and post-treatment data, assuming the distribution of data values will be similar to the existing data set. The statistical evaluation assumed equal numbers of soil samples will be collected before and after treatment. The formula used for sample size calculation, based on the two-sample T-test, is:

$$n = 2 \frac{s^2}{d^2} (z_{1-\alpha} + z_{1-\beta})^2 + \frac{z_{1-\alpha}^2}{4}$$

Where: s^2 = sample variance of log transformed data set for BaP
 δ = delta (the minimum detectable relative difference between the pre-treatment and post-treatment mean)
 $Z_{1-\alpha}$ = normal quantile for specified confidence level (from lookup table)
 $Z_{1-\beta}$ = normal quantile for specified power level (from lookup table)

A variety of assumed percent reductions were applied as δ values in the calculations to assess the affect of the size of δ on the sample requirements. Because it is usually desirable to achieve statistical confidence of at least 90 percent and power of at least 80 percent, α and β were set at 0.10 and 0.20, respectively. The results of the calculations are shown below:

<u>Percent Reduction</u>	<u>Number of Samples</u>
20	1,296
50	178
75	45
85	25
95	6

Based on the statistical calculations and in consideration of budgetary constraints, the objective for P1 will involve the collection of 45 soil samples at each pre- and post-treatment monitoring event, such that 75-percent reduction can be observed at 90 percent confidence and 80 percent power.

The sample estimates are high because of the large range of concentrations in the existing soil data set for

the MW-15 area. Reducing the level of confidence and power required would ease sample number requirements. For example, specifying a confidence of 80 percent and a power of 70 percent would produce a requirement of only 37 samples to observe a percent reduction of 50 percent, or 371 samples to observe a reduction of 20 percent.

As stated previously, the statistical simulation assumes that the pre- and post-treatability data will exhibit similar characteristics to the existing data set. This assumption is likely over-conservative. The pre-treatment soil samples will be selected from zones of highest contamination based on visual and olfactory evidence. It is anticipated that the pre-treatment soil concentrations will contain significantly fewer low and non-detected concentrations of COCs than the existing data set, and hence a smaller variance.

Therefore, the type of statistical analysis that will be conducted on the pre- and post-treatability data will likely be more powerful than indicated by the above calculations. Post-treatment samples will be collected from soil borings adjacent to the pre-treatment soil borings. This may allow for a natural pairing of pre- and post-treatment samples that will increase the statistical power of the analyses. Also, depending on the distribution of the sample data, other statistical tests that depend on distributional assumptions may be reasonable. In conclusion, the number of samples that will be collected to satisfy the primary objective may be conservative. Even if statistical verification of the contaminant reductions is not possible at the specified level of confidence and power, the size of the pre- and post-treatment data sets will allow for useful qualitative to semi-quantitative evaluations of Cool-Ox™ performance in support of objective P1.

3.1.2 Data Analysis and Interpretation of Results

The analytical results will be reduced through the calculation of summary statistics (for example, mean, median, minimum, maximum, standard deviation, and upper and lower confidence limits) for different subgroups of data, such as sample locations/borings, sampling events, and sample depths. Data mapping and visualization tools will also be used.

The null hypothesis, (H_0) is that the true mean contaminant concentration of the post-treatment sample population is less than or equal to 75 percent below the mean of the pre-treatment sample population. The alternative hypothesis (H_a) is that the true post-treatment mean is greater than 75 percent less than the pre-treatment mean. Statistical calculations to test the H_0 and H_a hypotheses will be performed at a minimum for the COCs.

The data will be evaluated for distributional assumptions using appropriate visual displays, such as probability plots and statistical techniques including the Shapiro-Wilk goodness of fit test. Based on the results of the distributional testing, parametric tests (for example, the t-test) or non-parametric tests will be selected to compare pre- and post-treatment means. Appropriate methods for the treatment of nondetect results will be used (EPA 2000), and the data will also be evaluated for outliers using an inferential method for outlier detection. If outliers are identified, one or all of the following will occur:

- Possible causes for the outliers will be investigated with the project team.
- Sample distribution and statistical testing will be performed with and without the outliers and both sets of findings will be interpreted.

The concentrations of COCs in soil samples collected before the treatability study will be evaluated to determine if spatial or statistical trends in the distribution of values are apparent. As discussed in Sections 3.1.1, post-treatment samples will be collected from soil borings immediately adjacent to the pre-treatment soil borings. This sampling approach will allow for a natural pairing that can be statistically evaluated. Depending on the spatial and statistical distribution of the data, other methods of data evaluation may be appropriate.

3.2 SECONDARY OBJECTIVES

Secondary objectives are not critical to the evaluation of the technology but should provide additional information helpful in understanding the capabilities of the technology. The following subsections discuss secondary objectives and the tasks to be conducted and data to be collected.

3.2.1 Secondary Objective S1

As discussed in Section 1.5, Cool-Ox™ reagent consists of solid peroxygens suspended in an aqueous matrix. The reagent reacts in the subsurface and generates hydrogen peroxide. Additionally, DTI will treat the Cool-Ox™ reagent with bromide. These factors will assist the SITE Program with evaluating the ability of the suspended peroxygens in penetrating the aquifer matrix.

The SITE Program will evaluate this objective by collecting groundwater samples at points that are located equidistant from the reagent injection points approximately 30 days after Cool-Ox™ treatment.

Locations of the proposed groundwater sampling points are presented on Figure 1-8. As discussed in Section 1.6.1, DTI may adjust the location of their injection points. This is inherent in the deployment methodology. Consequently, the proposed locations on Figure 1-8 may be revised to better evaluate this objective. Groundwater samples will be collected from depths that correspond to the mid point of the reagent injection intervals. At this time, the groundwater sample collection intervals are anticipated to be at depths of 12.5 and 7.5 ft. The samples will be analyzed for bromide and hydrogen peroxide.

Bromide is a conservative tracer and will begin to migrate with the prevailing hydraulic gradient as soon as it is injected into the aquifer. Most or all of the bromide will likely have migrated out of the treatment volume after 60 days. Likewise, DTI estimates that the reagent will cease to generate hydrogen 60 to 90 days after treatment.

The timing and placement of boring discussed in this section must be considered when evaluating this objective. With these factors in mind, the presence of bromide is a clear indication that the aqueous portion of the reagent penetrated to that portion of the aquifer, but not necessarily the reagent. The presence of hydrogen peroxide will be considered as a positive indication that the reagent penetrated to that portion of the subsurface. The absence of hydrogen peroxide will be considered an indication that the reagent was unable to penetrate to that portion of the aquifer or possibly that the reagent was spent by the time the sample was collected.

3.2.2 Secondary Objective S2

The vendor claims that the Cool-Ox™ process does not harm the native biota and will enhance the growth of native biological organisms. For this reason, the SITE Program will collect samples to evaluate the population of microorganisms and the changes over the course of the project.

Groundwater samples will be collected from monitoring wells MW-15 and MW-15A for seven days prior to treatment and at approximately 30 and 90 days after treatment. The samples will be analyzed for heterotrophic plate counts, PAH degraders, dissolved oxygen, NO₃, SO₄, Fe²⁺, Mn²⁺, total inorganic carbon, TOC, BOD, and COD.

Soil samples will be collected prior to Cool-Ox™ treatment and 30 and 90 days after treatment. The sample will be analyzed for heterotrophic plate counts and PAH degraders.

The results of the post-treatment samples will be compared to the pre-treatment results to evaluate the potential that the population of biological microorganisms increase, decrease, or remain the same with time. The analysis will focus on plate counts and PAH degraders. The chemical data that will be collected as part of this objective will be reviewed to support conclusions regarding the biological population.

3.2.3 Secondary Objective S3

The S3 objective is to evaluate the vendor claim that there is sufficient iron and other catalytic metals present in the subsurface for the technology to perform and the addition of iron or other additives is not necessary. This claim may be correct in native soils, but the MW-15 area is largely Fill and the amount of metals present in the Fill may help or hinder the process.

The 45 pre-treatment soil samples that the SITE Program will collect for evaluation of the P1 objective will be analyzed for metals. Groundwater samples will be collected from MW-15 and MW-15A on three occasions over a seven day period prior to injection of the reagent. The groundwater samples will be analyzed for metals. The results will be compared to ranges of metals that are typically found in naturally occurring soils and groundwater. The evaluation may shed light on the potential of the Cool-Ox™ process.

3.2.4 Secondary Objective S4

It is acknowledged by the demonstration participants that the groundwater contamination present at the site is a consequence of source areas contributing to the groundwater contamination. It is also acknowledged by the demonstration participants that this deployment of the Cool-Ox™ technology is not intended to fully remediate the site. The objectives of the vendor and the site owner are provided in Section 1.6. It is acknowledged by the demonstration participants that concentrations of contaminants in groundwater may be reduced by the technology, but the concentration may rebound to concentrations above the regulatory benchmark concentrations or to pre-treatment levels. One possible exception is

those wells in the immediate area of MW-15. For that reason, the SITE Program will monitor the concentrations of dissolved phase VOCs and SVOCs in the MW-15 area.

Baseline groundwater samples for analysis of VOCs and PAHs will be collected from monitoring wells MW-15 and MW-15A three times approximately seven days apart prior to treating the MW-15 area. Groundwater samples will be collected 30 and 90 days after treatment. The pre-treatment concentrations will be compared to post-treatment concentrations to evaluate the objective. Concentrations will be graphed with respect to time to evaluate the change in concentrations over the 90 day post-treatment period.

3.2.5 Secondary Objective S5

DTI claims that foaming of the reagent is an indicator of subsurface contamination and that this is taken into consideration when deploying the technology. Seven of borings that will be installed for the P1 objective will be collocated with Cool-Ox™ injection points. Thus, the SITE Program will have information on the subsurface conditions at those locations. The SITE Program will observe deployment of the technology in an attempt to understand some of the rationale behind the deployment process.

The observations will include documentation of the pattern of boring installation, locations of the foam mushrooms, injection intervals, and injection quantities. Documentation will include recording pertinent information in the field book and collection of photographs. The data gathered during the pre-treatment sampling coupled with the observations made during deployment of the technology may shed additional light on the potential of this technology.

3.2.6 Secondary Objective S6

DTI claims that a major advantage of the Cool-Ox™ technology is that the technology operates at a basic pH and that it doesn't generate heat. The SITE Program will collect temperature and pH measurements at monitoring wells MW-15 and MW-15A during the groundwater sampling events and for seven days after treatment is complete in the MW-15 area. Likewise, temperature and pH measurements will be collected from MW-13A after completion of treatment in the MW-13 area. Measurements will be collected from monitoring well MW-13B if the well is accessible.

3.2.7 Secondary Objective S7

DTI claims that the Cool-Ox™ reagent will generate surfactants which make contaminants more susceptible to dissolution and biodegradation, and more mobile. DNAPL that is more mobile can be more easily removed from the subsurface by extraction wells. This is the primary rationale for injecting the reagent in the MW-13 area near the three operating extractions wells.

The SITE Program will collect measurements of DNAPL recovery over approximately three weeks prior to injection in the MW-13 area. Measurements of the DNAPL column in monitoring well MW-13A and extraction wells EW-1, EW-2, and EW-3 will also be collected. These data will be compared to historical data that NewFields has collected as part of their routine monitoring of the system to determine if the measurements are consistent.

Post-treatment monitoring will consist of collection of DNAPL recovery measurements on a weekly basis for 90 days after injection of the reagent in the MW-13 area by NewFields as part of their routine monitoring of the system. The SITE Program will also take independent measurements during the 30 and 90 day post-treatment sampling events. Post-treatment recovery measurement will be compared to pre-treatment recovery measurements to determine if the treatment has enhanced DNAPL recovery.

3.2.8 Secondary Objective S8

DTI claims that the Cool-Ox™ reagent will generate surfactants which make contaminants more susceptible to dissolution and biodegradation and more mobile. The SITE Program will evaluate the mobility of the DNAPL recovered by the treatment system and from DNAPL that may be present in the extraction wells and monitoring wells MW-13A. Sampling will occur before Cool-Ox™ treatment and 30 and 90 days after treatment of the MW-13 area.

The samples will be analyzed for surface tension, density, and viscosity. One pre-treatment sample and two post-treatment samples from each location will be submitted for analysis of SVOCs, VOCs, and total petroleum hydrocarbon (TPH)-extractables. The contaminant analysis will provide information of the relative portions of contaminants that comprise the DNAPL and how that might change as a consequence

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of Cool-Ox™ treatment. The project chemist will review chromatograms, tentatively identified compounds, and raw laboratory data to identify compounds that may be indicative of surfactants that are generated by the technology.

4.0 SITE FIELD SAMPLING AND MEASUREMENT PROGRAM

The main objective of the planned SITE sampling and monitoring program is to provide sufficient data to allow EPA to evaluate the performance of the Cool-Ox™ technology and to meet the primary and secondary SITE demonstration objectives discussed in Section 1.7. The goal of the planned sampling program is to collect and analyze samples of sufficient number and quality that the results accurately reflect the performance of the treatment technology during the demonstration.

Several sampling objectives must be met for this demonstration to produce well-documented, defensible data that are of known and reproducible quality. The following items describe the general sampling objectives for the demonstration of the technology:

- Collect representative samples;
- Preserve and ship samples in a manner designed to ensure sample integrity and continued representativeness;
- Maintain proper chain-of-custody control of all samples, from collection to analysis; and
- Follow QA/QC procedures appropriate for EPA SITE Applied Research projects (EPA 1991).

The following sections discuss sample collection and preparation, field QC samples, field measurement procedures, and sample custody procedures.

4.1 SITE SOIL SAMPLING PROCEDURES

The SITE program will mobilize the necessary equipment (GeoProbe®) and personnel to advance 15 soil borings within the MW-15 treatment area. Planned soil boring locations were selected to evaluate the success of the Cool-Ox™ technology. Eight soil borings will be advanced equidistant between the Cool-Ox™ injection points, and seven planned soil borings will be collocated with the Cool-Ox™ injection points.

The soil borings will be advanced using a track- or truck-mounted GeoProbe® to an approximate depth of 15 ft bgs in the MW-15 treatment area. Three soil samples will be collected from each boring using a

Geoprobe® brand or equivalent dual-tube sampling system. Soil samples will be collected from above the Miller Creek aquitard. Soil samples will be collected in accordance with procedures specified in American Society for Testing and Materials (ASTM) D6282-98(2005) “*Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations.*”

Soil samples will be collected from within the MW-15 treatment area before and after the injection of the Cool-Ox™ oxidant. The pre-treatment borings will be located relative to fixed points at the site and the location carefully documented in the field book. Post-treatment soil boring will be located as close to the pre-treatment boring locations as practicable.

Soil samples for VOC analysis will be collected using an Encore® sampling device. Discrete soil samples will be collected directly from the soil cores from the most highly contaminated areas based on visual or olfactory observations. If an Encore® sampling device cannot be used due to adverse soil conditions (such as rocky, sandy, or moist soil), the SITE Program will collect the sample for VOC analysis by placing the soil directly in a 4-ounce jar with septum using a disposable polyethylene scoop. After collecting soil for VOC analysis, additional soil samples will be collected for SVOCs, metals, heterotrophic plate counts and PAH degraders. A summary of the analytical methods, minimum sample volumes, preservation requirements, and holding times for the soil samples are presented in Table 4-1.

The following information will be recorded in the field logbook for each soil sample:

- Sample interval or depth, where appropriate (in feet bgs)
- Sample recovery (inches)
- Unified Soil Classification System (USCS) soil type
- Physical description of the core sample

The physical description of the soil sample will include the following information:

- Texture, including features such as grain size, grain shape, and sorting
- Color
- Consistency, including features such as plasticity and bedding features

- Moisture content
- Depth at which the water table is encountered
- Additional observations such as odor, staining, presence of organic materials

Upon completion, each boring will be grouted with a sodium bentonite slurry from the base of the boring to the ground surface using a high-pressure grout pump. The grout will be continuously pumped through the rods filling the probe hole as the rods are removed to ensure the hole is properly sealed.

4.2 SITE GROUNDWATER SAMPLING PROCEDURES

The overall goal of groundwater sampling is to collect samples that are representative of conditions at the site. In order to meet the primary and secondary SITE demonstration objectives, groundwater samples will be collected from monitoring wells and from vertical profiling. The sampling methods are described in the sections that follow.

4.2.1 Monitoring Well Sampling

Low-flow purging and sample collection methods will be used to collect groundwater samples from monitoring wells. Low-flow sampling techniques maintain minimal flow through the native formation, filter pack, and well screen to minimize suspension of solids and loss of volatiles due to degassing (Puls and Barcelona 1996). Groundwater sampling procedures will be as follows:

- The breathing zone will be monitored with a photo ionization detector (PID) meter during removal of the well cap
- The well will be opened, and allowed to equilibrate
- The water level and total depth within the well casing will be measured and recorded. If floating product is present, its thickness will be measured with an oil/water interface probe
- Continuous discharge pumps with variable flow-rates with dedicated Teflon® or polyethylene tubing will be used to purge and sample the well. The pump intake will be placed at the midpoint of the screen
- The discharge end of the tubing will be connected to a flow-through cell module, and the well will be purged at 500 milliliters per minute or less. Drawdown in the well will be monitored and will not be allowed to exceed 0.33 ft of drawdown

- The purged water will be monitored for pH, conductivity, temperature, turbidity, dissolved oxygen, and oxidation-reduction potential. The parameters will be recorded at 3-minute intervals until stabilization occurs or 3 well casing volumes have been removed
- The well will be considered stable when three successive readings are within ± 0.1 pH unit, $\pm 3\%$ conductivity, ± 10 mv for ORP, and $\pm 10\%$ for turbidity and dissolved oxygen
- When the well has stabilized or three well volumes have been purged, the discharge hose will be disconnected from the flow-through cell and the purge rate lowered to 100 milliliters or less. One sample system volume will be purged at the lower rate
- The sample containers will be filled directly from the purging hose
- The samples will be labeled, preserved, and placed in a cooler with ice

Data on pumping rates, drawdown, purge volumes, and water quality parameters will be recorded in field logbooks. Purge water will be containerized for later disposal in the on-site treatment system.

A summary of the analytical methods, minimum sample volumes, preservation requirements, and holding times for the groundwater samples are presented in Table 4-1.

4.2.2 Vertical Profile Sampling

Depth-discrete groundwater sampling (referred to as vertical profiling) will be conducted in the MW-15 treatment volume following the injection of the Cool-Ox™ reagents. Thirteen borings will be advanced using a GeoProbe® to obtain groundwater samples at depths of 7.5 ft and 12.5 ft below the ground surface. These sample depths correspond to the center of the Cool-Ox™ injection intervals above the Miller Creek aquitard.

Depth-discrete groundwater sampling will be conducted using a sealed grab sampler with a Geoprobe® brand or equivalent dual-tube sampling system to prevent cross-contamination. A sealed sampling device consisting of a well screen housed within a protective sheath to which are attached an expendable drive point, drive rod(s), and drive head will be used. The sampler is initially driven with the outer casing in place. Rubber O-rings keep the device water tight, eliminating the threat of formation fluids entering the screen before deployment and assuring sample integrity. The sampler will be pushed to the desired sampling depth, and a probe rod will be used to knock out an expendable drive point, and the outer casing

will be retracted to expose the screen to formation water. The formation water will be allowed to equilibrate with atmospheric conditions prior to sampling. A groundwater sample will be collected using a peristaltic or other small diameter pump through a tube positioned within the screened area. After the sample is collected, the sampler will be advanced to the next corresponding sampling depth and the process repeated.

Upon completion, each boring will be grouted with a sodium bentonite slurry from the base of the boring to the ground surface using a high-pressure grout pump. The grout will be continuously pumped through the rods filling the probe hole as the rods are removed to ensure the hole is properly sealed.

Data on pumping rates, purge volumes, and water quality parameters will be recorded in field logbooks. Purge water will be containerized for later disposal in the on-site treatment system.

Depth-discrete groundwater samples will be analyzed for bromide and hydrogen peroxide. Hydrogen peroxide will be analyzed in the field using a Hach Kit. A summary of the analytical methods, minimum sample volumes, preservation requirements, and holding times for the depth-discrete groundwater samples are presented in Table 4-1.

4.3 SITE DNAPL MONITORING AND SAMPLING PROCEDURES

DNAPL recovered at the site is a dark viscous material. The viscous sticky nature of the DNAPL has fouled interface probes during historical measurement events. This method of measurement is not recommended by NewFields.

The DNAPL will be measured in the 1,000 gallon holding tank. The dimensions of the tank are such that an increase of one inch in height of DNAPL is equivalent to 16.3 gallons. A dedicated clean measuring tape will be lowered through the access hole on the top of the tank to the bottom of the tank. The tape measure will be withdrawn and the thickness of the DNAPL interval will be determined by the length of the product coating the tape. The value will be recorded to the nearest tenth of an inch. The previous measurement will be subtracted from the current measurement to determine the increase in DNAPL height since the previous measurement. The increase in DNAPL height will be multiplied by 16.3 to

determine the gallons of DNAPL recovered during the week. NewFields will use these same procedure to collect routine DNAPL recovery measurements on a weekly basis.

DNAPL will be measured in the monitoring and extraction wells by lowering a weighted rope to the bottom of the well. The rope will be allowed to soak in the DNAPL for approximately one minute. The rope will be withdrawn and the length of rope that is stained with the DNAPL will be measured and recorded to the nearest inch. The rope will be discarded with the personal protection equipment. A new rope will be used for each measurement.

If sufficient quantities of DNAPL are present, the SITE program will collect DNAPL samples from the DNAPL holding tank, monitoring well MW-13A and extraction wells EW- 1, EW-2 and EW-3 using disposable/dedicated, double check-valve bailers. The bailer will be lowered to the bottom of the tank or well and allowed to fill for approximately one minute. The bailer will be withdrawn and the DNAPL placed in the appropriate sample containers. If the volume in the bailer is not sufficient to fill the sample containers, the procedure will be repeated until the sample containers are full or no more product can be recovered.

A summary of the analytical methods, minimum sample volumes, preservation requirements, and holding times for the DNAPL samples are presented in Table 4-1.

4.4 FIELD OVERSIGHT

The SITE Program will conduct field oversight of the Cool-Ox™ injection process in the MW-15 treatment area. DTI claims that foaming of the reagent is an indicator of subsurface conditions and that it is an important tool for assisting them with deploying the technology. For this purpose, seven of the 15 borings that will be installed for the P1 objective will be collocated with Cool-Ox™ injection points.

The SITE Program will observe deployment of the technology in an attempt to understand some of the rationale behind the deployment process. The observations will include documentation of the pattern of boring installation, locations of the foam mushrooms, injection intervals, and injection quantities.

Documentation will include recording information in the field logbook and collection of photographs. The data gathered during the pre-treatment sampling coupled with the observations made during deployment of the technology may shed additional light on the potential of this technology.

4.5 QUALITY CONTROL SAMPLES

Field QC samples will be collected to evaluate the validity of samples collected during the demonstration. Field QC samples for this demonstration will include field duplicates, matrix spike and matrix spike duplicate (MS/MSD), and equipment blanks. Table 4-2 summarizes the types of field QC samples to be collected for soil, groundwater, and DNAPL samples. The following sections describe the field QC samples and how they will be used to evaluate the validity of the field sampling effort.

Field Duplicate Samples

Field duplicate samples are two samples collected at the same time and from the same source that are submitted as separate samples to one laboratory for analysis. Collection and analysis of field duplicates allows evaluation of the consistency of the overall sampling and analytical system.

The SITE program will collect field duplicate samples for every 5 percent of the soil samples and every 10 percent for groundwater samples collected during this demonstration. Duplicate samples will be analyzed for the same parameters as the environmental samples. The sampling location will be recorded in the field logbook, but duplicates will not be identified by the sample labeling. Field duplicates will be collected at randomly selected locations.

Analytical results from field duplicate samples will be used to evaluate precision by calculating the relative percent difference (RPD). Limits for precision have not been determined for solid matrices. A significant variance is commonly associated with soil duplicates because it is difficult to collect truly homogenous soil samples (EPA 1999). For this reason, field duplicate RPD will not be a critical QA/QC parameter with regard to usability of the data set.

Equipment Blanks

Equipment blank samples are samples of clean analyte-free water passed through and over the sampling equipment. These blanks permit evaluation of equipment decontamination procedures and potential cross-contamination of environmental samples between sampling locations. The SITE program will collect equipment blank samples for every 5 percent of the soil samples and every 10 percent for groundwater samples collected during this demonstration.

The equipment blank will be collected by pouring deionized water over or through the sampling equipment and collecting it in the appropriate sample containers. The blank will be analyzed for the same parameters as the environmental samples.

Trip Blanks

Trip blanks are analyzed to estimate incidental or accidental VOC contamination of the environmental samples during sampling, storage, and transportation to the laboratory. Trip blanks will be provided by the analyzing laboratory whenever samples for aqueous VOC analysis will be collected. The trip blank will accompany the sample containers from the laboratory until they are returned. Trip blanks will be analyzed for VOCs only.

MS/MSD Samples

MS/MSD samples are analyzed to evaluate the precision and accuracy of an analytical method for a particular environmental sample matrix. The MS sample is prepared by adding a known concentration of target analytes to an aliquot of the field sample. The specific compounds that will be selected for spiking will be representative of the range of compounds anticipated to be present in the samples from the Ashland MGP site and will specifically include some of the COCs considered critical to the primary demonstration objective. EPA and the analytical laboratory will agree on the final list of compounds to be used for spiking before sample analyses commence. To minimize errors, samples will be spiked when they are prepared for analysis at the laboratory. The MS/MSD samples measure the efficiency of all of the steps of the analytical method in recovering target analytes from an environmental sample matrix.

The SITE program will collect one MS and MSD sample for every 5 percent of soil samples and every 10 percent for groundwater samples collected during this demonstration. MS/MSD samples will be analyzed for the same parameters as the environmental samples. MS/MSD samples will be collected at locations to be selected in the field. If the results of MS/MSD analyses indicate that the percent recoveries or relative percent difference (RPD) are outside the established acceptance limits, appropriate laboratory and data validation protocols specific to the method will be followed to evaluate the usability of the data.

4.6 SAMPLE CUSTODY PROCEDURES

This section describes standard custody procedures for samples collected for chemical analysis for this project. These procedures will be used to maintain and document sample integrity during collection, transportation, storage, and analysis. These procedures include maintaining field notes and logbooks, sample identification and labeling, use of custody seals, chain-of-custody records, sample shipping, and cooler receipt.

4.6.1 Field Notes and Logbooks

The SITE program will record in ink all information pertinent to the sampling and measurement program in a consecutively-numbered bound field logbook. The information will be entered into the field logbook at the time of sampling. At a minimum, the logbook will contain the following:

Background Information

- Date and time of the sampling activities
- Personnel on site
- Weather conditions
- Purpose of sampling

Chronology of Sampling

- Description of sampling points and sampling methodology

- Number and volume of samples collected
- Date and time of collection
- Sample identification number
- Field observations about any problems encountered and deviations from the final QAPP

Sample Distribution

- Sample distribution and method of transport (name of laboratory where samples were sent, overnight courier service used, airbill number, and other information)
- Signature of sampler or field sample custodian

Each page will be dated and signed by the person making the entries. Logbooks are accountable field documents and serve as a chronological representation of the sampling and measurement program. Sufficient detail will be included in the logbook to provide a summary of sampling and measurement activities without relying on the recorder's memory.

4.6.2 Sample Identification and Labeling

Each sample container will be labeled with a unique sample identification number. The label will also identify the sampling location, date, time of collection, and analyses to be performed. The following sample numbering system will be used for this demonstration:

XX/ZZ/MMDDYYYY

Where:

XX = Sample Location

ZZ = Sample depth or depth range, if applicable (for example, 7.5 ft or 3-5 ft bgs)

MM = Month of sampling (for example, 10 for October)

DD = Date of sampling (for example, 04)

YYYY = Year of sampling (for example, 2006)

For example, sample number **SB1/3-5/10042006** represents a soil sample collected from SB1 location, at a depth range of 3 to 5 feet below the ground surface, on October 04, 2006.

Field duplicate samples will be designated by adding the number “9” to the sample location. For example, sample number **SB19/3-5/10042006** represents a duplicate soil sample collected from SB1 location, at a depth range of 3 to 5 feet below the ground surface, on October 04, 2006.

Sample type abbreviations which may be used include:

- SB – Soil Boring Sample
- GW – Ground Water Sample
- MW – Monitoring Well Sample
- EW – Extraction Well Sample
- TS – Treatment System Sample
- TB – Trip Blank
- EB – Equipment Blank
- FD – Field Duplicate
- MS/MSD – Matrix Spike/Matrix Spike Duplicate Sample

The identification label will be completed with the following information written in indelible ink:

- Project name
- Sample identification number
- Date and time of sample collection
- Analyses required

4.6.3 Sample Custody and Shipment

The field team will follow appropriate chain-of-custody procedures for each sample from the time it is collected. Samples will be retained at all times in the field crew's custody until shipment. The field crew will ship samples to the laboratory at the end of each day or sampling event as appropriate for the required sample holding times. Sample custody will begin when the samples are placed into a cooler or other appropriate container in the possession of the designated field sample custodian. A line item on the

field chain-of-custody report form will be completed and initialed by the field sample custodian. The following information will be completed on the chain-of-custody form:

Project No.:	Enter the complete project number
Project Name:	Enter "Cool-Ox Demo"
Name of sampler:	Enter the name of the person collecting the samples
Sample destination:	Enter the name of the laboratory performing the analyses
Sample Number:	Enter the sample ID number
Date:	Enter the date of sample collection
Time:	Enter the time of sample collection
Number/type of containers:	Enter the number and type of containers
Sample Description/Type:	Enter the sampling location and type
Analysis Required:	List the parameters to be analyzed and QC requirements (MS/MSD)
Preservatives used:	Enter the type of preservatives for each sample
Airbill number:	Enter the FedEx airbill number
Signatures:	Enter signatures of individuals involved in custody transfer, including date and time of transfer
Remarks:	Enter remarks related to sample analysis, such as samples selected for MS/MSD analysis

When all line items are completed or when the samples are picked up, the SITE program will sign and date the chain-of-custody form, list the time, and confirm that all descriptive information contained on the form is complete.

All samples will be packaged and labeled for shipment in compliance with current regulations. Only metal or plastic ice chests will be used. Ice chests used to ship aqueous samples will be lined with two plastic bags; the plastic bags around the aqueous samples will be sealed by twisting the top and securely taping the bag closed to prevent leaks. The drain holes inside the chests will be taped shut. Styrofoam, bubble wrap, or other packing materials will be used to absorb shock. Chain-of-custody record forms and any other shipping and sample documentation will accompany the shipment. These documents will be enclosed in a waterproof plastic bag and taped to the underside of the cooler lid. A temperature blank will

be included in each ice chest. Each ice chest prepared for shipment will be securely taped shut. Reinforced or other suitable tape (such as duct tape) will be used and wrapped at least twice around the ice chest near each end where the hinges are located. Sample shipping containers will be marked in accordance with U.S. regulations. A minimum of two custody seals will be placed on the sample-shipping containers.

When selecting means of shipping samples, field personnel will ensure that the method chosen will not cause the sample to exceed allowable holding times. When commercial common carriers are used to ship samples, all samples will be shipped for overnight delivery to the appropriate laboratory(s).

The laboratory sample custodian or designated alternate will receive and assume custody of samples until the samples have been properly logged in the laboratory and stored in a secured area. When a sample shipment is received at the laboratory, the shipping container will be inspected for warning labels and security breaches before it is opened. The sample custodian will open the container and carefully check the contents for evidence of breakage or leaking. Preservation requirements regarding temperature will be verified, as appropriate, for aqueous samples at the time samples are received. Deviations will be reported to the SITE program project manager immediately and will be noted in the monthly case narrative report.

The contents of the container will be inspected for chain-of-custody record forms and other information or instructions. The person making the entry will record the date and time on the chain-of-custody record form. The sample custodian will verify that all information on the sample container labels is correct and generally correlates with the information on the chain-of-custody record form, and will sign for the chain-of-custody record form. The chain-of-custody record form will be retained in the project file and a copy returned to the SITE program to verify receipt.

Any discrepancy between the samples and the chain-of-custody information, any broken or leaking sample bottles, or any other abnormal situation will be reported to the laboratory project manager. The SITE program will be informed of any problem, and corrective action options will be discussed and implemented. The problem and its resolution will be noted in a corrective action memorandum, which will be initialed and dated by the sample custodian. Each shipment of samples received at the laboratory

will be assigned a work order number. Each sample in the shipment will be given a unique laboratory sample number that includes the work order number and an identifying code. A laboratory sample label specifying the unique identifier will be attached to each container. The work order will specify the samples to be analyzed, the analysis required, the level of QC requested for the demonstration, and any other necessary information. The work order will be given to group leaders, who will schedule extractions and analyses to meet applicable holding times. Bench sheets, initiated at the first point of sample preparation, are to accompany the samples throughout the analytical sequence. For most analyses, sample preparation data and analytical results are entered into computer spreadsheets that generate both analytical report forms and QC summary forms.

4.7 EQUIPMENT DECONTAMINATION

Pre-cleaned, disposable (one-time use) sampling equipment will be used where possible to minimize equipment decontamination requirements. Reusable monitoring and sampling equipment such as water-level indicators, interface probes, submersible pumps, and Geoprobe® well screens will be decontaminated between sampling points in the following manner:

1. Wash with low-phosphate detergent (Alconox® or equivalent)
2. Generous rinse with potable water
3. Distilled and deionized water rinse
4. Allow to air dry

Geoprobe® equipment requiring will be decontaminated between borings using a high-pressure, hot-water spray. The decontamination station will be set up at a designated area on the site. All fluids and solids generated from equipment decontamination will be captured and containerized. Following use, disposable sampling equipment will be containerized. At the end of the project, the Geoprobe® will be decontaminated by high-pressure, hot-water spray.

The outside surfaces of containers used to collect samples for chemical analysis may also require decontamination prior to shipping to the analytical laboratory. When necessary, the sealed sample containers will be cleaned using an Alconox® detergent wash and tap-water rinse in the personnel

decontamination station. Personal decontamination equipment and procedures in the contaminant reduction zone are described in the site-specific HSP in Appendix A.

4.8 MANAGEMENT OF INVESTIGATION-DERIVED WASTE

Investigation derived waste (IDW) generated during this demonstration will include soil boring material, purge water, disposable soil-sampling equipment, decontamination fluids, and discarded personal protection equipment (PPE). All soil boring material, disposable soil-sampling equipment, and decontamination fluids will be considered potentially contaminated and will be containerized in labeled drums. Solids will be containerized separately from liquids, and all drums will be labeled with date/time/contents/contact information. Discarded personal PPE will be placed in plastic garbage bags and labeled. Purge water will be pumped into carboys and discharged to the on-site treatment system.

5.0 ANALYTICAL PROCEDURES AND CALIBRATION

Analytical methods, calibration, and sample archiving requirements are described in this section. The discussions of analytical methodology are based largely on information supplied by the analytical laboratories.

5.1 SELECTION OF ANALYTICAL METHODS AND LABORATORIES

In selecting appropriate analytical methods for this demonstration, the specific analyte of interest, sample matrix, and minimum detectable concentrations needed to achieve project objectives were considered. The selection process used the following hierarchy of analytical method references:

1. EPA-approved methods described in the following references:

- \$ Methods for the Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, EPA 600/4-79-020, 1979, Revised March 1983.
- \$ Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, U.S. Environmental Protection Agency, SW-846, Third Edition, 1986 with 12/96 Updates.

2. Widely used reference methods such as those found in the following references:

- \$ Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, American Public Health Association, American Water Works Association and Water Environment Federation.
- \$ Methods published annually by the American Society for Testing and Materials (ASTM).
- \$ Other methods approved by EPA for use in the SITE Program.

In addition, the capabilities of the laboratories were considered when selecting the methods to be used. Table 4-1 summarizes the methods chosen to prepare and analyze the samples collected during the technology.

5.2 ANALYTICAL METHODS

This section briefly describes the analytical methods that will support the primary objective of the demonstration, and notes any project-specific requirements for these reference methods as cited in Table 4-1. The methods that support the primary objective P1 include those that will measure the COCs in soil.

Methods that support secondary objectives will be performed in accordance with the reference methods as documented and amended by the laboratories' internal standard operating procedures (SOPs). No project-specific requirements over and above the standard method and laboratory procedures are established in this QAPP for methods that support secondary objectives.

5.2.1 Volatile Organic Compounds

Concentrations of VOCs will be determined using gas chromatograph/mass spectrometer (GC/MS) in accordance with EPA Methods 8260B. This method will focus on the reporting of COCs related to the petroleum hydrocarbon contamination at the site, which include the BTEX parameters. The laboratory will also report tentatively identified compounds (TICs) in accordance with SW-846 guidance to identify classes of chemicals produced by the treatment process, and will provide total ion current chromatograms for inspection by the project team as part of the data deliverables. The laboratory will perform TIC mass spectral library searches for every chromatographic peak that is greater than 20 percent of the height of the nearest internal standard.

If COCs are present at varying concentrations, such that dilution of a sample is required to bring the concentration of some COCs within the calibration range of the analytical system, the laboratory will also analyze the undiluted sample to achieve project-required reporting limits (PRRL) for other COCs. If analysis of an undiluted sample will overload the analytical system, the laboratory will analyze the sample at the lowest dilution possible to achieve the lowest possible reporting limit for all COCs. Results from all dilutions will be reported by the laboratory.

5.2.2 Semivolatile Organic Compounds

Concentrations of SVOCs will be determined using GC/MS in accordance with SW-846 Method 8270C. The laboratory will also report TICs in accordance with SW-846 guidance to identify classes of chemicals produced by the treatment process, and will provide total ion current chromatograms for inspection by the project team as part of the data deliverables. The laboratory will perform TIC mass spectral library searches for every chromatographic peak that is greater than 20 percent of the height of the nearest internal standard.

If COCs are present at varying concentrations, such that dilution of an extract is required to bring the concentration of some analytes within the calibration range of the analytical system, the laboratory will also analyze the undiluted extract to achieve the lowest possible reporting limit for the other COCs. If analysis of an undiluted extract will overload the analytical system, the laboratory will analyze the extract at the lowest dilution possible to achieve the lowest possible reporting limit for all COCs. Results from all dilutions will be reported by the laboratory.

5.3 CALIBRATION PROCEDURES FOR ANALYTICAL SYSTEMS

Calibration procedures and frequencies will be in accordance with the listed EPA reference methods. Calibration standards will be prepared from standard reference materials.

For the GC/MS methods that support the primary objective (VOC and SVOC methods), calibrations will be performed with a minimum of five standards. The concentration of one standard will be at or below the PRRL. In all cases, the concentration of calibration standards will define the working range of the instrument system. Results outside the calibration range will be reported, but will be flagged.

Each calibration standard will be analyzed by the same technique used to introduce samples into the GC. Instrument response will be used to prepare a calibration curve for each analyte. If the coefficient of correlation (r) for each compound is ≥ 0.99 , the linearity of the calibration is considered sufficient to proceed with sample analyses. Further, instrument response will be used to calculate a Response Factor (RF) for each compound, relative to the response of an Internal Standard. The relative standard deviation (RSD) will be calculated for the RFs for each compound. If the RSD is ≤ 30 percent, linearity through the origin can be assumed, and the average RF will be used in place of a calibration curve.

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The calibration curve will be verified every 12 hours of operation by analyzing a mid-point calibration standard. If the percent difference between the known and measured concentrations of the calibration verification standard is ≤ 20 percent, the multi-point calibration will still be considered valid, and analysis of samples can proceed. If the mean RF is used in place of a calibration curve, the calibration will be verified by calculating the RF from the calibration verification standard. If the percent difference between the mean RF from the calibration and the RF from the calibration verification standard is ≤ 30 percent, the multi-point calibration will still be considered valid, and analysis of samples can proceed.

6.0 QUALITY ASSURANCE OBJECTIVES AND QUALITY CONTROL REQUIREMENTS

This section discusses QA objectives and QC requirements for the laboratory analytical data that will be generated for the COCs and other chemical and physical parameters. The establishment of and compliance with QA/QC requirements are most crucial for analytical data that will support the primary objectives.

6.1 QUALITY ASSURANCE OBJECTIVES

The overall QA objective for this evaluation is to produce well-documented data of known quality. Quality is measured by monitoring data precision and accuracy, completeness, representativeness, comparability, and reporting limits for the analytical methods. The objectives for these parameters are discussed in Sections 6.1.1 through 6.1.5.

If analytical data fail to meet the QA objectives described in this section (for example, because of matrix interferences), EPA will explain the reason in the Technology Evaluation Report (TER), and will describe the limitations and usefulness of the data in the TER. The following corrective actions may be taken for data that do not meet QA objectives: (1) verify that the analytical measurement system was in control, (2) thoroughly check calculations, (3) use data qualifiers (flags), and (4) re-prepare and reanalyze the affected samples if authorized by the EPA WAM and if a sufficient quantity of sample is available.

6.1.1 Precision and Accuracy

Precision and accuracy goals depend on the types of samples and analyses and the ultimate use of the analytical data. Table 6-1 summarizes the precision and accuracy goals for COCs for soil and groundwater, respectively. Equations used to estimate precision and accuracy are given in Section 6.2.

To generate MS/MSD samples in the laboratory, two aliquots of selected samples will be spiked. A spiking solution concentration and amount will then be identified for addition to the two aliquots taken for MS/MSD analysis (see Section 6.3.2.1). Laboratory control samples and laboratory control sample

duplicates (LCS/LCSD) will also be prepared and analyzed to determine whether poor MS recovery is due to matrix effects or laboratory methods.

6.1.2 Representativeness

For this project, representativeness involves sample numbers, sampling locations, and sample collection and preparation methods, including the collection of field QC samples. As described in Sections 3.0 and 4.0, the evaluation plan has been designed to ensure that (1) a sufficient number of samples will be collected at select locations, and (2) each sample will be collected in a manner that ensures maximum representativeness.

6.1.3 Completeness

Completeness is an assessment of the amount of valid data obtained from a measurement system relative to the planned level of data collection. The percent completeness is calculated by the number of samples yielding acceptable data divided by the total number of samples planned to be collected and multiplied by 100 (see Section 6.2.4). The QA objective for the degree of completeness is 75-90% percent (see Table 6-1). This objective is for sampling of soil, groundwater, and DNAPL before and after injection of Cool-Ox™. If the completeness value is less than the stated value for the parameter, EPA will document why this objective was not met and the impact of a lower percentage, if any, on the project.

6.1.4 Comparability

The comparability of the data will be maximized by using standard EPA analytical methods, when possible. The planned methods are specified, and deviations from the methods will be documented in the ITER. Results for COCs will be reported in standard units as shown in Table 6-1. Laboratory calibrations will be based on standards traceable to National Institute of Standards and Technology (NIST). Comparability will also be maximized through the use of consistent sample collection techniques and analytical methods throughout the evaluation.

6.1.5 Reporting Limits

PRRLs for COCs and other target analytical parameters are listed in Table 6-1. These reporting limits are based on the laboratory's experience with the analytical method and similar matrices as well as method detection limit studies conducted annually by the laboratory. These reporting limits are based on undiluted samples. Actual reporting limits may be higher where samples with elevated concentrations require dilution for analysis.

Reporting limits may be a concern for this project because individual compound concentrations are expected to vary significantly within a sample class. Analytical sensitivity and data quality could also be impacted by the high overall levels of hydrocarbons detected in some samples. Where appropriate, the samples will be analyzed twice at two different dilutions to expand the applicable quantitation range. The SITE Program will work with the laboratory to assess whether re-analysis with additional cleanup steps might improve sensitivity and data quality for critical samples. Results that are less than the laboratory's reporting limits and above the method detection limits will be flagged to indicate the greater uncertainty associated with such values.

6.2 CALCULATION OF DATA QUALITY INDICATORS

This section presents the specific calculations that will be used to describe the following data quality indicators for the COCs: precision, accuracy, representativeness, completeness, and detection limits.

6.2.1 Precision

Precision will be estimated by analyzing duplicate MS samples or duplicate samples. The RPD between the analyte levels measured in the MS/MSD samples will be calculated using the following equation:

$$RPD = \{ |MS - MSD| / [0.5 * (MS + MSD)] \} * 100$$

The RPD between the analyte levels measured with LCS/LCSD samples will be calculated as:

$$RPD = \{ |C_O - C_D| / [0.5 * (C_O + C_D)] \} * 100$$

Where:

C_o = the original sample concentration

C_D = the duplicate sample concentration

6.2.2 Accuracy

Accuracy will be estimated by calculating the percent recovery of laboratory MS/MSD samples using the following equation:

$$\%Rec = [(C_j - C_o) / C_i] * 100$$

Where:

%Rec = Percent recovery

C_j = Measured concentration in spiked sample aliquot

C_o = Measured concentration in unspiked sample aliquot

C_i = Actual concentration of spike added

Percent recovery for LCS/LCSD samples will be calculated using the following equation:

$$\%Rec = [(C_j / C_i) * 100$$

Where:

%Rec = Percent recovery

C_j = Measured concentration in the LCS

C_i = Actual concentration of LCS

Accuracy goals are presented on Table 6-1

6.2.3 Representativeness

Representativeness will be reviewed in relation to the sampling design. Field duplicate samples and equipment blanks will also be used to assess representativeness for groundwater samples.

6.2.4 Completeness

Completeness will be reported as the percentage of measurements judged valid. The following equation will be used to determine completeness:

$$\%C = (V / T) * 100$$

Where:

%C = Percent completeness

V = Number of measurements judged valid

T = Total number of measurements

6.3 INTERNAL QUALITY CONTROL REQUIREMENTS

Internal QC consists of checks and procedures to ensure that QA objectives are met. These checks and procedures document compliance with the objectives or demonstrate the need for corrective action.

These checks are of two kinds: (1) monitoring field activities, such as sample collection and shipping; and (2) monitoring laboratory activities, such as extraction and analysis. These checks are discussed in the following sections.

6.3.1 Field Quality Control Checks

Field QC checks will be conducted to determine the quality of field activities, including sample collection, handling, and shipment. In general, these QC checks will assess the representativeness of the samples and ensure that the degree to which the analytical data are representative of actual site conditions is known and documented. Field QC checks will consist of equipment blanks and field duplicate samples.

Rinsate Blanks

Rinsate blanks will be used to check the potential of cross-contamination of samples. Cross-contamination potentially may occur during sampling or handling if decontamination procedures are

inadequate. These blanks will be analyzed for COCs using the methods described previously.

If contamination is found in a rinsate blank, the laboratory will determine whether the contamination originated in the field or during analysis in the laboratory by preparing and analyzing a laboratory blank. Contamination is defined as a positive result greater than the method detection limit or a negative result (interference) of greater magnitude than the PRRL. If the laboratory concludes that the contamination occurred during laboratory analysis, the laboratory will implement corrective action to find the source of contamination and will reanalyze the equipment blank (and potentially all associated samples) if sufficient volume exists. If it is determined that contamination originated in the field, the Tetra Tech project manager will be immediately notified and the data will be reevaluated to determine its usefulness.

Field Duplicate Samples

Field duplicate samples will be used to check the reproducibility of sample collection. These duplicate samples will be analyzed in the same manner as the primary samples. The analytical results of the primary sample and the duplicate sample will be compared. If the analytical results of the duplicate sample and primary sample have a RPD within plus or minus 35 percent, field sample collection procedures will be considered adequate. However, the 35 percent RPD criterion will be considered advisory and not a QC limit.

6.3.2 Laboratory Quality Control Checks

Laboratory QC checks are designed to determine analytical precision and accuracy, demonstrate the absence of interferences and contamination from glassware and reagents, and ensure the comparability of data. Laboratory QC samples consist of LCS/LCSD, method blank, MS/MSD, and duplicate sample analyses. Individual methods may contain additional QC checks. In addition, the laboratory will complete initial calibrations and calibration verification checks. Laboratory internal QC checks for the analytical methods are summarized in Table 6-2. Table 6-2 also indicates the required frequency, acceptance criteria, and corrective actions for each QC check. Each of these checks and their frequencies are discussed in the following sections.

MS/MSD

MS samples will be used to assess the accuracy of the analytical method. They will be prepared by spiking a field sample with standard reference materials at a known concentration. The MS will be prepared and analyzed in the same manner as field samples throughout the entire analytical process. MS results outside acceptance criteria may be caused by a matrix effect associated with the sample or by laboratory practices not meeting specifications. To determine whether the excursion is due to sample matrix effects, LCS/LCSD samples will be analyzed. Frequency, acceptance criteria, and corrective action for MS samples are presented in Table 6-2.

Duplicate Analyses

Duplicate analyses will be used to determine the precision of the analytical results. They will be conducted by analyzing a selected field sample in duplicate. Frequency, acceptance criteria, and corrective action for duplicate samples are presented in Table 6-2.

Laboratory Control Sample/Laboratory Control Sample Duplicate

Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) samples will be prepared by spiking a well-characterized matrix similar to the sample matrix with standard reference materials at a known concentration. The materials used for the LCS/LCSD will be from a source different from the source of the calibration standards. LCS/LCSD samples will be used to verify laboratory accuracy in the absence of chemical matrix interferences related to field samples. The LCS/LCSD will be prepared and analyzed in the same manner as field samples throughout the entire analytical process. Frequency, acceptance criteria, and corrective action for LCS/LCSD samples are presented in Table 6-2.

Surrogate Compounds

Surrogate compounds will be used as a measure of accuracy in each sample analyzed by GC (TPH) or GC/MS (VOCs and SVOCs). Surrogate compounds are compounds that are similar in nature to the analytes of interest, but rarely found in the environment. By adding known amounts of these compounds

to each sample prior to preparation and analysis, recovery of each through the analytical process can be calculated. This recovery can then be used as a surrogate measure of how well analytes of interest might have been recovered. Acceptance criteria and corrective action for surrogate compounds are presented in Table 6-2.

Internal Standard Areas

Changes in internal standard (IS) areas will be used to monitor change in GC/MS system sensitivity over time. By comparing the area of each IS in the daily calibration verification standard to its area in subsequent samples, a significant increase or decrease in system sensitivity can be detected. Acceptance criteria and corrective action for IS areas are presented in Table 6-2.

Method Blanks

Method blanks are used to identify system or process interferences or contamination of the analytical system that may lead to reporting of biased results. Method blanks will consist of reagent water (containing reagents specific to the method) that is carried through the entire analytical procedure, including preparation and analysis. The method blank will be prepared and analyzed in the same manner as field samples throughout the entire analytical process. Frequency, acceptance criteria, and corrective action for method blanks are presented in Table 6-2.

7.0 DATA REDUCTION, VALIDATION, AND REPORTING

Correct equations and procedures must be used to ensure that all data generated and processed are valid, defensible, and comparable. The following sections describe the data reduction, validation, and reporting procedures that will be used in this evaluation.

7.1 FIELD DATA REDUCTION AND REPORTING

Field data will be generated during this project. The field manager will record all field data in bound logbooks, along with any associated calibration checks, assumptions, deviations, or anomalies. All entries will be dated and initialed by the personnel collecting the data. The data will then be transcribed to spreadsheets or databases and evaluated as described. Field data will be reviewed by the project chemist to assure that correct data reduction procedures were used and that there are no transcription errors.

7.2 LABORATORY DATA REDUCTION AND REPORTING

This section will discuss laboratory data reduction, data validation, and laboratory data reporting requirements.

7.2.1 Laboratory Data Reduction

The analytical methods to be used for this demonstration contain detailed instructions and equations for calculating compound concentrations and other parameters. The analysts responsible for the measurements will enter raw data into logbooks or onto data sheets. Data will be reduced to the units specified in Table 6-1 using the procedures in the analytical methods. All numerical results will be reported as calculated without censoring for any laboratory reporting limits. Numerical results falling below the reporting limit and above the method detection limit will be reported for all sample and QC sample analyses; however, these data will be flagged with a designation determined by the laboratory.

7.2.2 Laboratory Data Validation

Individual analysts will review the data generated each day to determine the need for corrective action or rework. Data reviewed will include calibration and QC data. Individual analysts will also review data for completeness. The data will also undergo a second review by a laboratory supervisor independent of the data generation effort. This second review is typically conducted within several days after the data are generated. Laboratory logbooks and notebooks will also be reviewed on a monthly basis by the laboratory supervisor.

The Tetra Tech project chemist will be responsible for post-laboratory validation of laboratory data. This will include a full review of the case narrative, QC summary forms against project requirements, and other QC criteria. In addition, a full data validation will initially be conducted on 10 percent of the laboratory data. If significant deficiencies are identified in the 10 percent validation effort, the entire data package will be fully validated. The Tetra Tech project chemist will conduct a technical review of data received from the analytical laboratory. In addition to a review of the case narrative and comparison of QC summary forms to project requirements, the technical review will also include the following:

- \$ Method Compliance
- \$ Holding Time compliance
- \$ Calibration
- \$ Field and Laboratory Blank Results
- \$ Spike Recoveries
- \$ Field and Laboratory Duplicate Results
- \$ Internal Performance Standards
- \$ Other Laboratory QC (per Method)

Analytical outlier data are defined as QC data lying outside a specific QA objective range for precision or accuracy for a given analytical method. If QC data are outside control limits, the laboratory supervisor will investigate to determine the probable causes of the problem. If necessary, the sample will be reanalyzed, and only the reanalyzed results reported. If the problem is with the matrix, both initial and

reanalyzed results will be reported and identified in the laboratory report. If reanalysis is not feasible, the initial analysis results will be reported and the results will be flagged and discussed in the laboratory report.

Suspected project outlier data will initially be identified as sample data that are of questionable validity because they are (1) outside specified acceptance limits established around the central tendency estimator of the relevant data set, or (2) otherwise grossly dissimilar from expected results. For data that are known or assumed to be normally distributed, the initial identification criterion will be the 99 percent confidence limit defined by the Student's two-tailed t-distribution test. Potential outlier data will be assessed on a case-by-case basis for physical or analytical anomalies. Only if there is clear evidence of an anomaly will the data be considered project outlier data. Project outlier data will be identified and reported in the final laboratory report, but will not be used to determine overall project results.

7.2.3 Laboratory Reporting Requirements

The Tetra Tech project manager and the Tetra Tech project chemist will approve the completed laboratory report before it is used to prepare the Innovative Technology Evaluation Report (ITER) and Technology Evaluation Report (TER). The laboratory will submit full analytical and QC reports to include the following, as appropriate:

- Case narrative including a list of samples reviewed with field names and laboratory names cross-referenced, discussion of any deviations from the QAPP and any other nonconformance and the associated corrective actions, discussion of any analytical or procedural problems encountered and corrective actions, and an explanation of the data qualifiers used
- Completed chain-of-custody forms
- Sample result summary forms for all samples, field QC samples, and method blanks
- Spreadsheet containing any positive or negative results that are less than the method detection limit. These data will not be qualified or flagged but will bear a laboratory disclaimer as to the limits of the data usability
- QC summary forms for MS/MSD samples, LCS, and sample log-in sheets
- True Value® summary form containing the concentrations for the initial calibration verification (ICV) standard and continuing calibration verification (CCV) standard
- Raw data sheets and quantitation reports for initial calibration results, ICV and CCV results, and

blank results

- Sample preparation logs and run logs

In accordance with standard document control procedures, the laboratory will maintain on file original copies of all data sheets and logbooks containing raw data, signed and dated by the responsible analyst. The laboratory will maintain separate instrument logs to enable the run sequences to be reconstructed for individual instruments. The laboratory will maintain all data on file for 5 years in a secure archive warehouse accessible only to designated laboratory personnel. The data will be disposed of in the interim only after instructions to do so have been received from Tetra Tech or EPA.

7.3 PROJECT DATA REPORTING

The compilation of data from this technology evaluation project will be reported in an ITER. The content and format of the ITER will be similar to that shown in the following outline:

1.0 INTRODUCTION

1.1 PROJECT BACKGROUND

- 1.1.1 Site History
- 1.1.2 Site Description
- 1.1.3 Technology Description
- 1.1.4 Technology Application

1.2 EVALUATION OBJECTIVES AND APPROACH

1.3 KEY CONTACTS

2.0 TREATMENT EFFECTIVENESS

2.1 EVALUATION PROCEDURES

- 2.1.1 Evaluation Preparation
- 2.1.2 Evaluation Design
- 2.1.3 Sampling and Analytical Methods
- 2.1.4 Quality Assurance and Quality Control Program
 - 2.1.4.1 Field Quality Control Program
 - 2.1.4.2 Laboratory Quality Control Checks

2.1.4.3 Field and Laboratory Audits

2.2 EVALUATION RESULTS AND CONCLUSIONS

- 2.2.1 Primary Objectives
- 2.2.2 Secondary Objectives
- 2.2.3 Data Quality
- 2.2.4 Conclusions

3.0 TECHNOLOGY APPLICATIONS ANALYSIS

3.1 FEASIBILITY STUDY EVALUATION CRITERIA

- 3.1.1 Overall Protection of Human Health and the Environment
- 3.1.2 Compliance with Applicable or Relevant and Appropriate Requirements
- 3.1.3 Long-Term Effectiveness and Permanence
- 3.1.4 Reduction of Toxicity, Mobility, or Volume Through Treatment
- 3.1.5 Short-Term Effectiveness
- 3.1.6 Ability to Implement
- 3.1.7 Cost
- 3.1.8 State Acceptance
- 3.1.9 Community Acceptance

3.2 TECHNOLOGY APPLICABILITY

3.3 KEY FEATURES OF THE TREATMENT TECHNOLOGY

3.4 MATERIALS HANDLING REQUIREMENTS

3.5 SITE SUPPORT REQUIREMENTS

3.6 LIMITATIONS OF THE TECHNOLOGY

3.7 STATUS OF THE TECHNOLOGY

4.0 ECONOMIC ANALYSIS

4.1 INTRODUCTION

4.2 CONCLUSIONS

4.3 ISSUES AND ASSUMPTIONS

4.4 BASIS FOR ECONOMIC ANALYSIS

- 4.4.1 Site and Facility Preparation Cost
- 4.4.2 Permitting and Regulatory Costs

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- 4.4.3 Equipment Costs
- 4.4.4 Start-up and Fixed Costs
- 4.4.5 Labor Costs
- 4.4.6 Consumables and Supplies Cost
- 4.4.7 Utilities Costs
- 4.4.8 Effluent Treatment and Disposal Costs
- 4.4.9 Residuals and Waste Shipping, Handling, and Transport Costs
- 4.4.10 Analytical Costs
- 4.4.11 Facility Modifications, Repair, and Replacement Costs
- 4.4.12 Site Restoration Costs

5.0 REFERENCES

APPENDIX A - VENDOR CLAIMS

APPENDIX B - CASE STUDIES

8.0 ASSESSMENTS

Measurement systems and associated data will be assessed on a day-to-day basis by project staff (A routine assessments@), and as periodic, rigorous investigations by independent technical experts (A audits@). Corrective actions will be formulated and implemented in response to any data quality issues raised during routine assessments or audits.

8.1 ROUTINE ASSESSMENTS

On a routine basis, the entire project team, including the project manager, field staff, QA staff, and subcontracted laboratories, will assess measurement systems and identify data quality issues as they arise. Identification of data quality issues will be based on the following:

- Assessments of equipment adequacy or condition for the intended measurement
- Any difficulties involved in implementing the sampling and analytical methods identified in the QAPP
- Comparison of actual results to what is reasonably expected
- Evaluation of the internal consistency of results
- Comparison of QC results to acceptance criteria or QA objectives
- Any other evidence uncovered during day-to-day operations that measurement systems are not in control or that data are of questionable validity

Routine assessments are often the most effective in identifying data quality issues as they arise. However, personnel directly and intimately involved in a project may not always recognize when a data quality issue has arisen. Therefore, audits will be conducted to provide an independent view of measurement systems and data, and to provide additional assurance that data quality issues are identified and appropriately corrected.

8.2 AUDITS

QA audits are independent assessments of measurement systems and associated data. QA audits may be internal or external and most commonly incorporate activity-by-activity reviews of all critical measurement systems (a A technical systems audit,@ or TSA). Personnel independent of the sampling and

analytical teams conduct internal audits. An independent organization, typically EPA, conducts external audits.

8.2.1 Internal Audits

At the request of the WAM, an internal TSA of field sampling and measurements systems will be conducted during the initial phases of the field sampling associated with the demonstration. At a minimum, the following activities will be audited during the field TSA:

- \$ Sample collection
- \$ Sample preparation
- \$ Process measurements
- \$ Field QA and QC
- \$ Field documentation and chain-of-custody
- \$ Decontamination
- \$ Sample labeling, packaging, and shipping
- \$ Project management/QA management of quality-affecting activities

At the request of the WAM, Tetra Tech also will conduct a TSA of each subcontract laboratory with respect to each primary measurement. Specifically, laboratories conducting analysis of COCs will be audited.

For laboratory TSAs, both compliance with the EPA-approved planning document and with reference methods identified in the planning document will be audited. The following specific activities will be audited at a minimum:

- \$ Sample receipt/sample storage
- \$ Internal chain-of-custody procedures
- \$ Sample preparation
- \$ Sample extraction
- \$ Sample analysis
- \$ Standards preparation, storage, and use of second source standards

- \$ Calibration
- \$ QC procedures
- \$ Data reduction, validation, and reporting
- \$ Project management/QA management of quality-affecting activities

All field and laboratory TSAs performed by Tetra Tech will be conducted by a technical expert designated by the Tetra Tech SITE QA Manager. The Tetra Tech SITE QA Manager will ensure that each auditor is sufficiently removed organizationally from project activities to provide an independent assessment. Auditors may be either Tetra Tech employees or consultants and will be assigned prior to the initiation of measurement activities.

TSAs will be conducted in accordance with Tetra Tech's internal guidance for SITE projects and with EPA/SITE technical directives and guidance. Based on Tetra Tech's internal guidance, the audit process to be implemented by the assigned auditor can be summarized as the following sequential steps:

1. A checklist is developed from the EPA-approved QAPP and from reference methods identified in the QAPP.
2. Actual project activities are observed and compared to the activities described in the EPA-approved QAPP and in the prescribed reference methods using the pre-prepared checklist.
3. Nonconformance and corrective actions are discussed on site; any immediate corrective action is observed and documented when possible.
4. A draft TSA report is prepared documenting any observed nonconformance, as well as any immediate corrective action that was implemented.
5. The draft TSA report is reviewed by the Tetra Tech SITE QA manager or his designee for technical and editorial correctness.
6. The draft TSA report is distributed to the auditee, the EPA WAM, the EPA QA Manager, and the Tetra Tech project manager and Tetra Tech SITE QA manager.
7. Any response to the draft TSA report is reviewed to assess the issue or proposed corrective action.
8. A final TSA report is prepared, subjected to internal review, and distributed to the auditee, the EPA WAM, the EPA QA Manager, and the Tetra Tech project manager and Tetra Tech SITE QA manager.

8.2.2 External Audits

TSA's of sampling and analysis procedures may also be conducted by EPA, or a designated EPA contractor, at the discretion of the EPA WAM and EPA QA manager. Most commonly, EPA will audit Tetra Tech's field sampling and measurement activities. If EPA elects to perform a field TSA, Tetra Tech will endeavor to coordinate the above-described internal TSA with the EPA TSA and to schedule the audits on sequential days. The internal TSA will then be identified as a pre-audit and will be used to identify issues for resolution during the EPA TSA. Alternatively, the EPA QA Manager may elect to simply provide oversight of the Tetra Tech internal TSA and to provide additional comments for input to the Tetra Tech audit report. Tetra Tech will include the EPA WAM in the debriefing for internal TSA's and will provide all documentation from internal TSA's to EPA for review.

Performance audits of critical analytical procedures may also be conducted by Tetra Tech or by EPA through issuance of blind QC samples that incorporate selected critical analytes or representative compounds for analysis by the subcontracted laboratories. However, no performance audits are currently planned for this project.

8.3 CORRECTIVE ACTIONS

Each member of the Tetra Tech project team is responsible for noting when routine assessments reveal that any field or laboratory measurement activity is (1) not in compliance with the EPA-approved QAPP, or (2) demonstrates a potential data quality issue. Each team member is further responsible for initiating a nonconformance memorandum or otherwise communicating the issue to the laboratory project manager or the Tetra Tech project manager, as appropriate. The nonconformance memorandum should document the problem, the probable impact on the quality of the associated data, and the immediate corrective actions implemented. To ensure that appropriate corrective action is implemented, copies of all nonconformance memoranda initiated by the field team shall be forwarded to both the Tetra Tech project manager and the Tetra Tech SITE QA manager, and all nonconformance memoranda initiated by laboratory personnel shall be forwarded to the laboratory project manager and QA manager, who will determine whether the nonconformance has been corrected appropriately and whether consultation with the Tetra Tech project manager is needed (the Tetra Tech project manager should be consulted for all

nonconformance that could significantly impact project data). The Tetra Tech project manager will then consult with the Tetra Tech SITE QA Manager and with the EPA QA Manager, as needed, to develop an appropriate plan of corrective action.

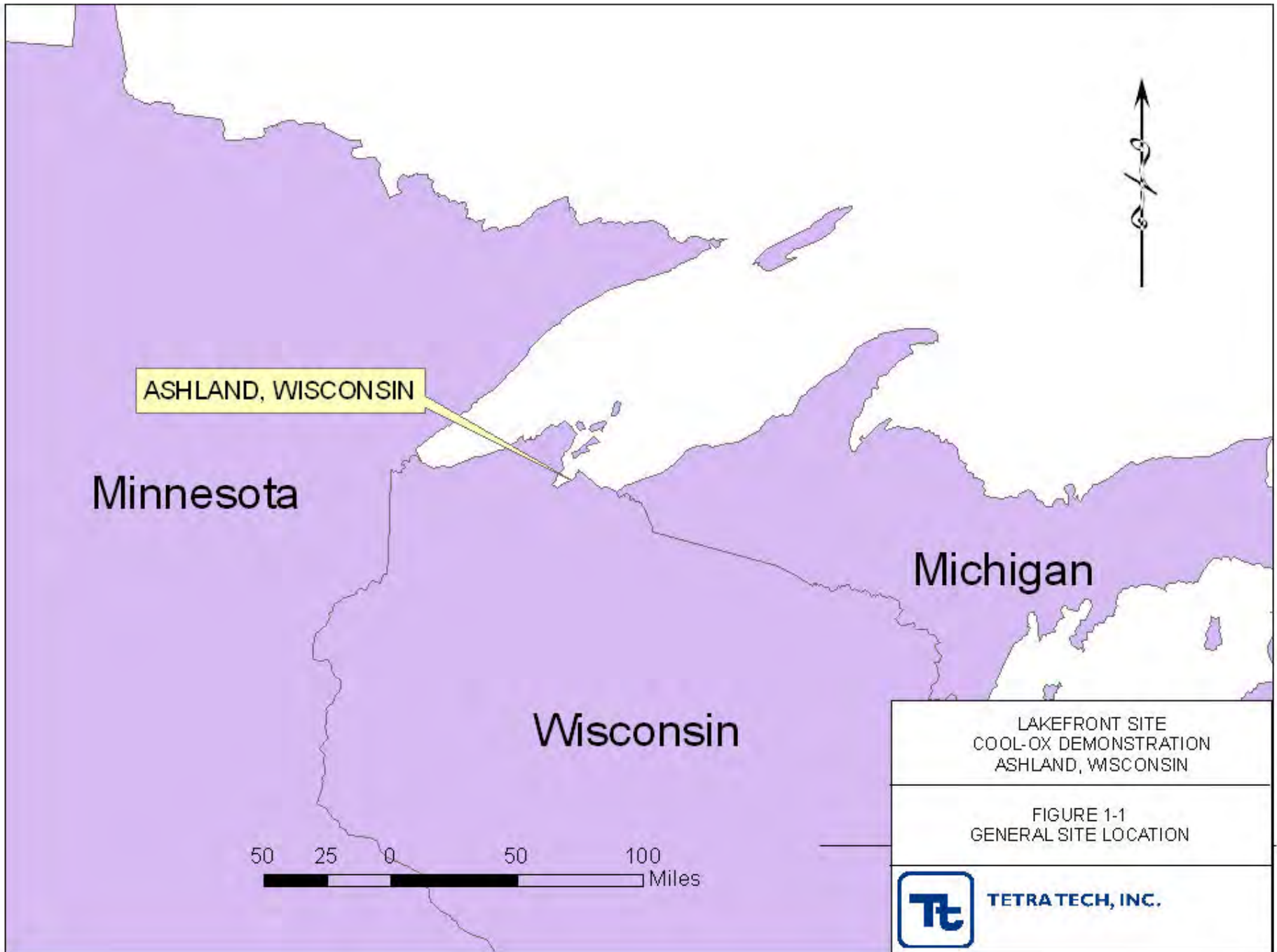
Once the corrective action has been identified, a corrective action memorandum or other appropriate documentation will be initiated by the Tetra Tech project manager or the laboratory project manager. The corrective action memorandum will document the corrective action, the personnel involved in the decision-making, and the personnel responsible for implementing the corrective action.

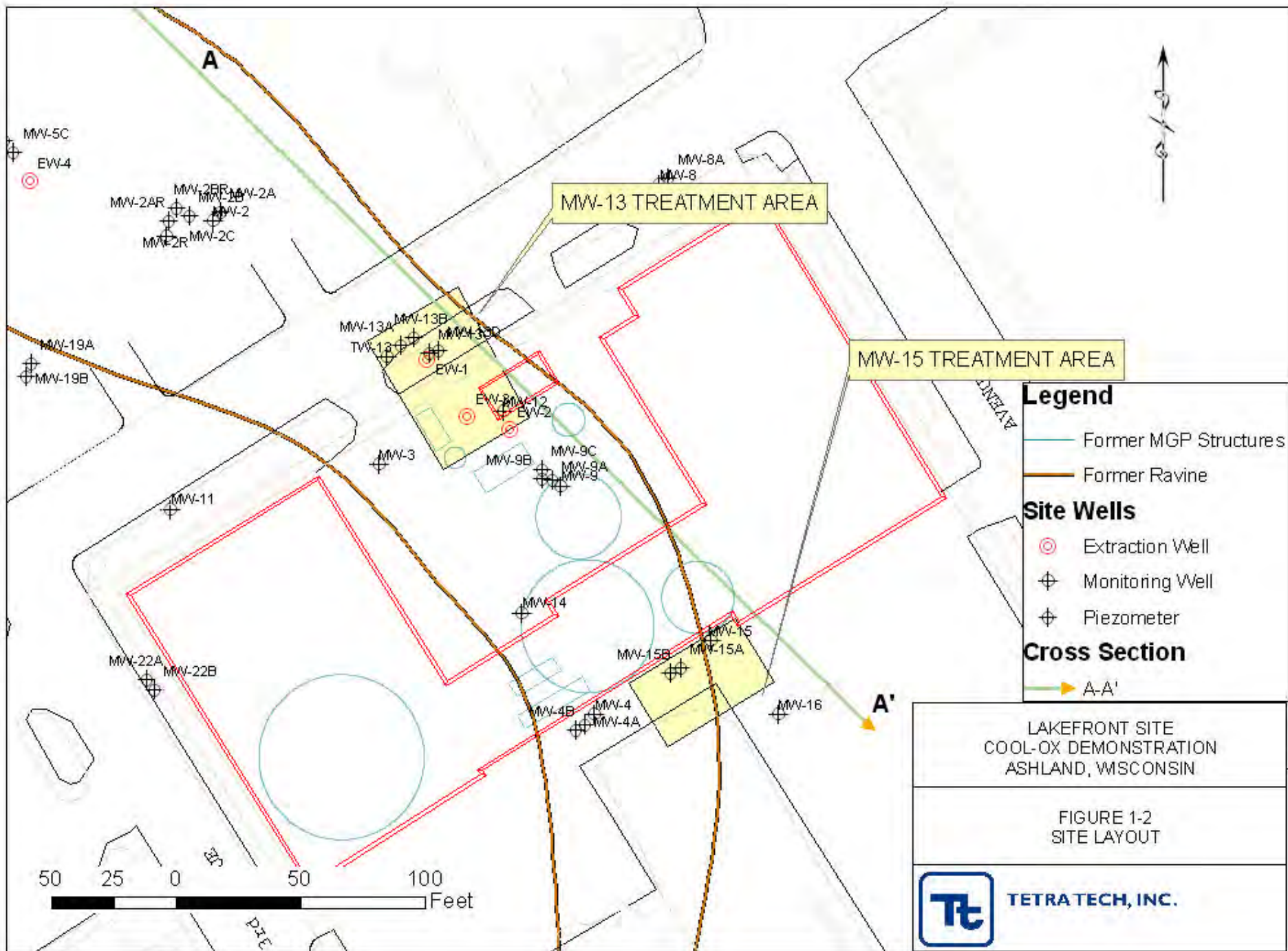
The Tetra Tech project manager will retain a copy of all nonconformance and corrective action memoranda generated by Tetra Tech project staff. Copies of all laboratory nonconformance memoranda and corrective action memoranda will be retained by the laboratory project manager and then included in the laboratory's final data report. For field nonconformance, the Tetra Tech project manager is directly responsible for ensuring that appropriate corrective action has been taken. For laboratory nonconformance, the laboratory project manager is responsible for ensuring that appropriate corrective action has been implemented and for keeping the Tetra Tech project manager informed of the status of laboratory corrective actions. For corrective actions taken in response to audits, the audit report and response serves as the documentation of the nonconformance and the corrective action. The Tetra Tech project manager and laboratory manager are responsible for ensuring that corrective actions identified through the audit process are completely implemented for field and for laboratory activities, respectively.

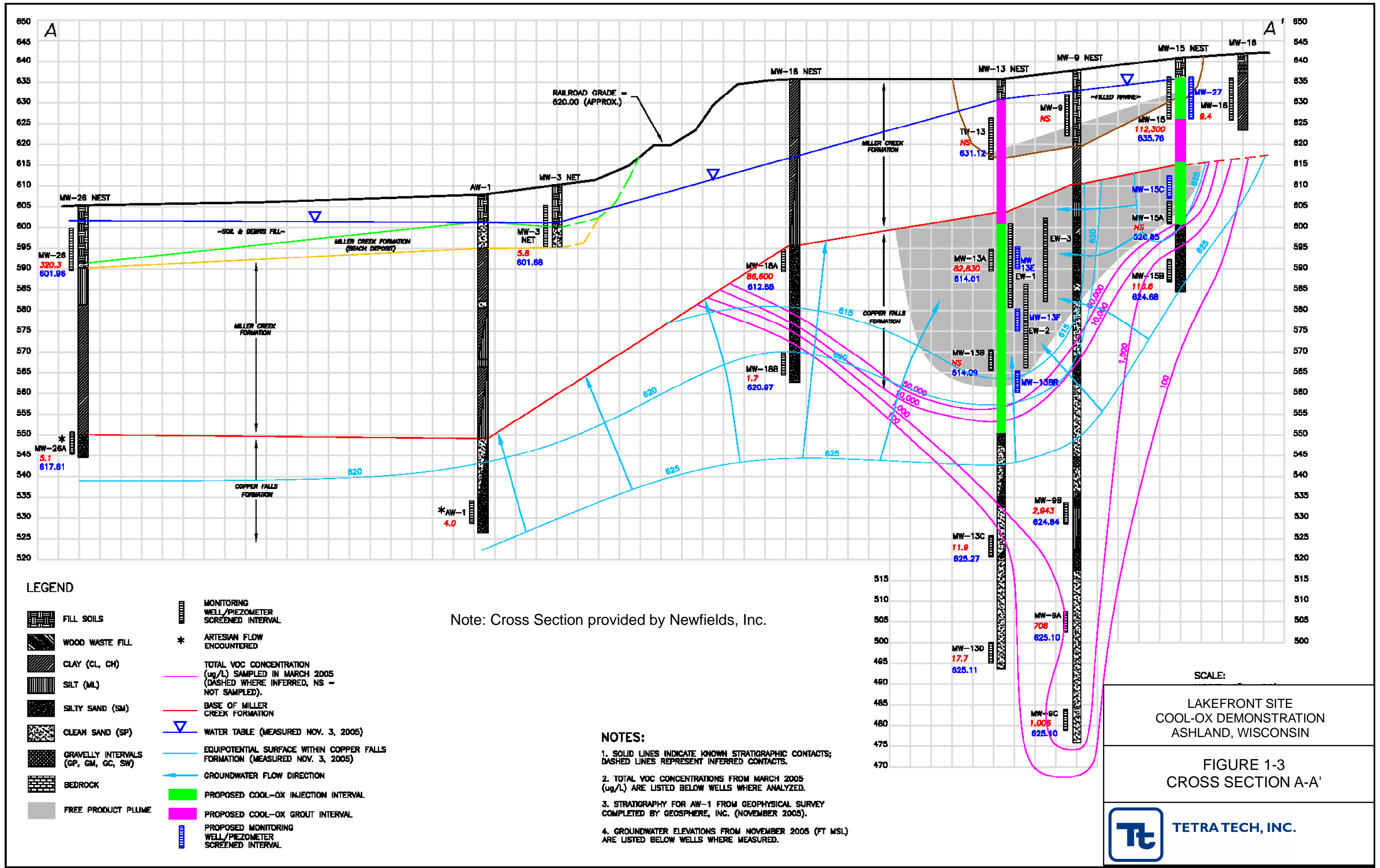
9.0 REFERENCES

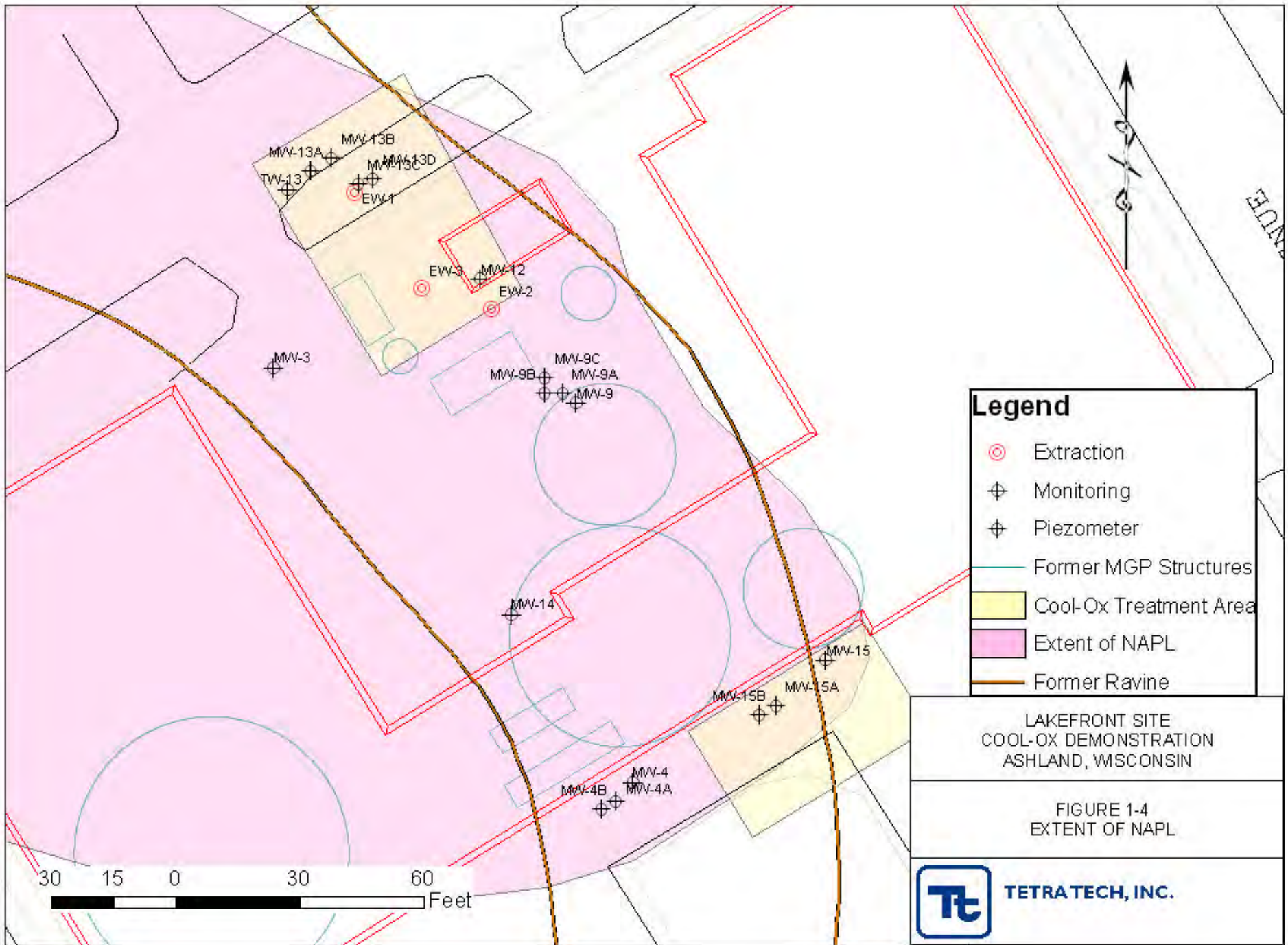
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FIGURES
(9 Pages)









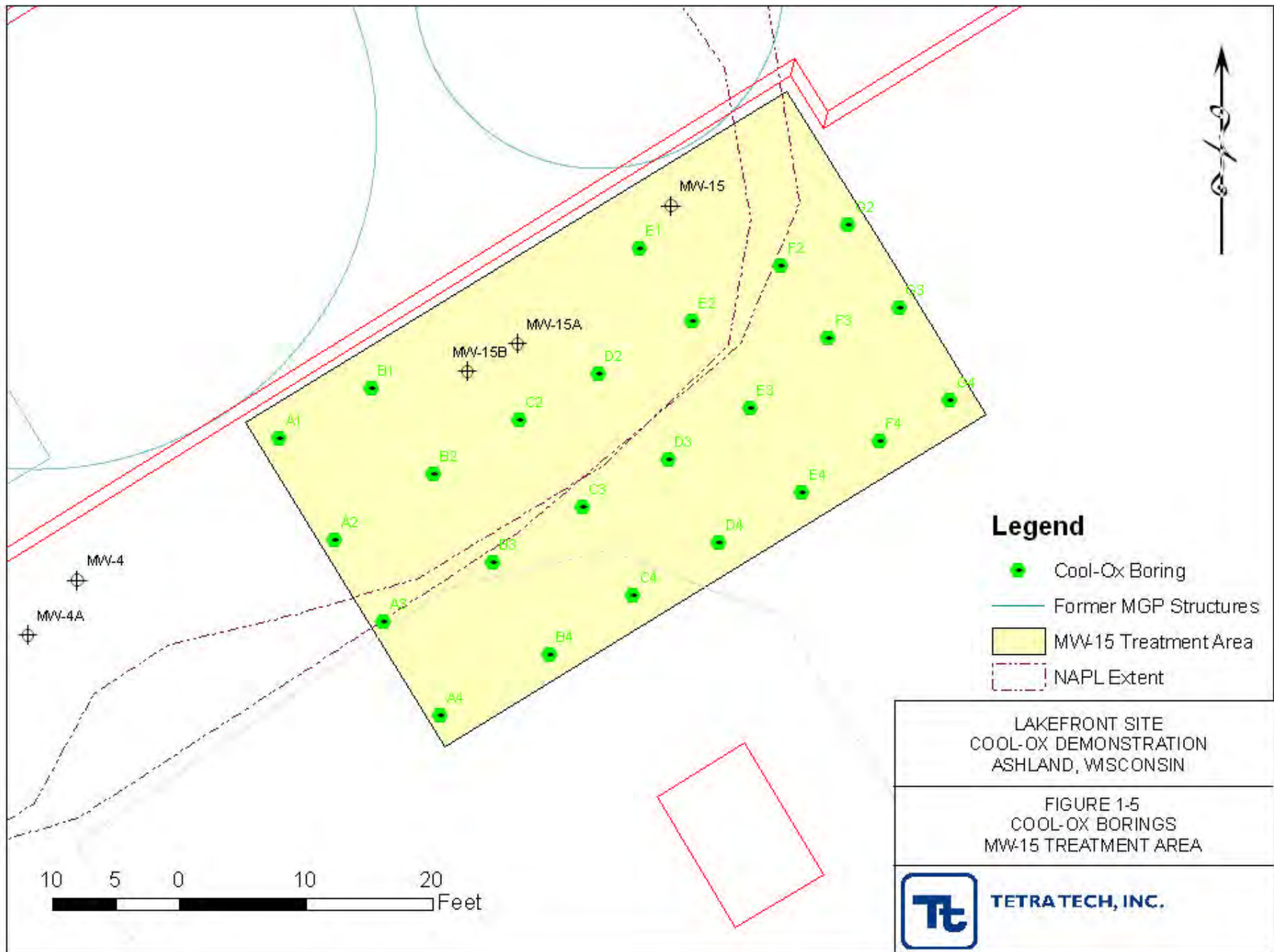
Legend

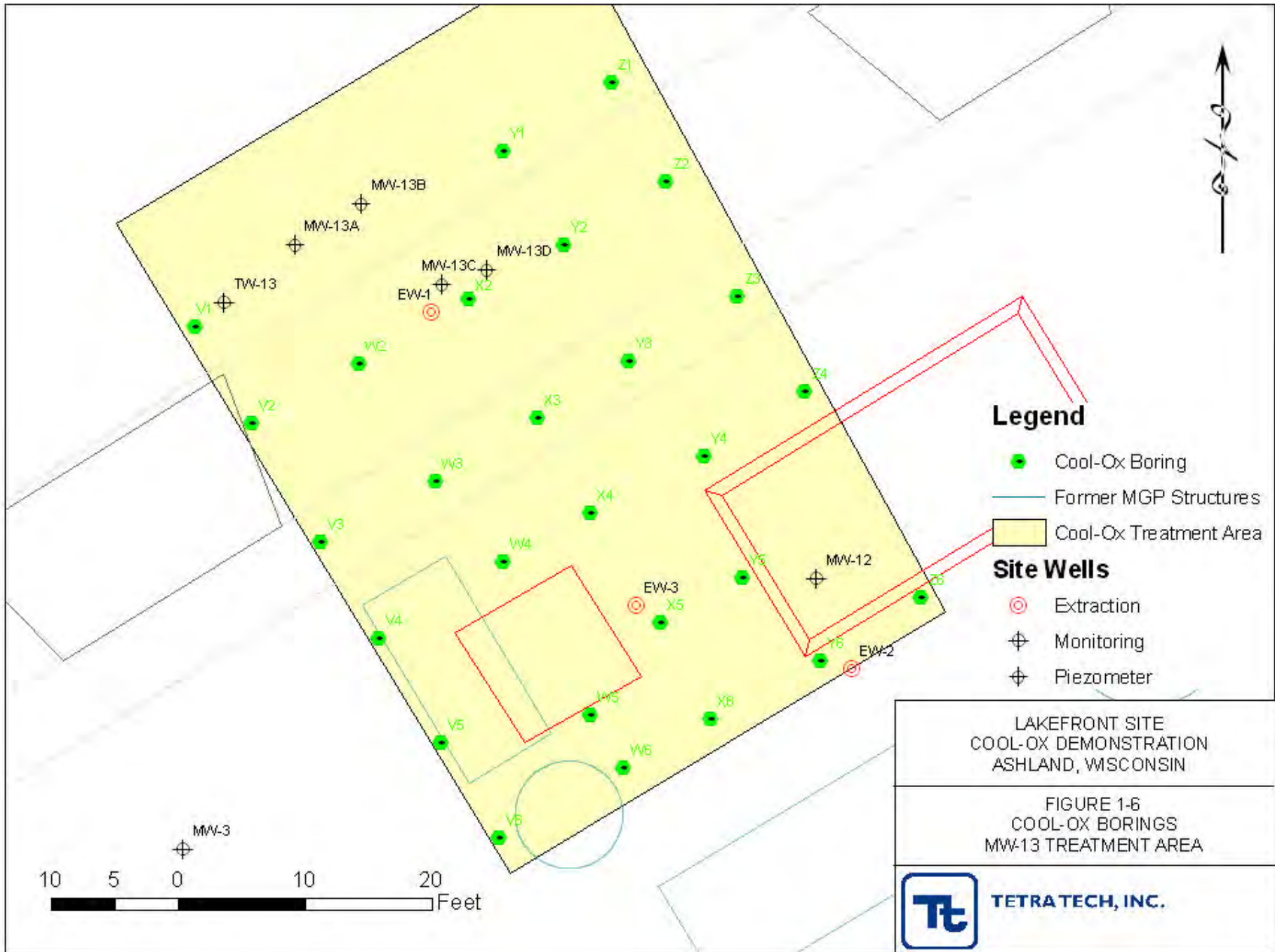
- ⊙ Extraction
- ⊕ Monitoring
- ⊕ Piezometer
- Former MGP Structures
- Cool-Ox Treatment Area
- Extent of NAPL
- Former Ravine

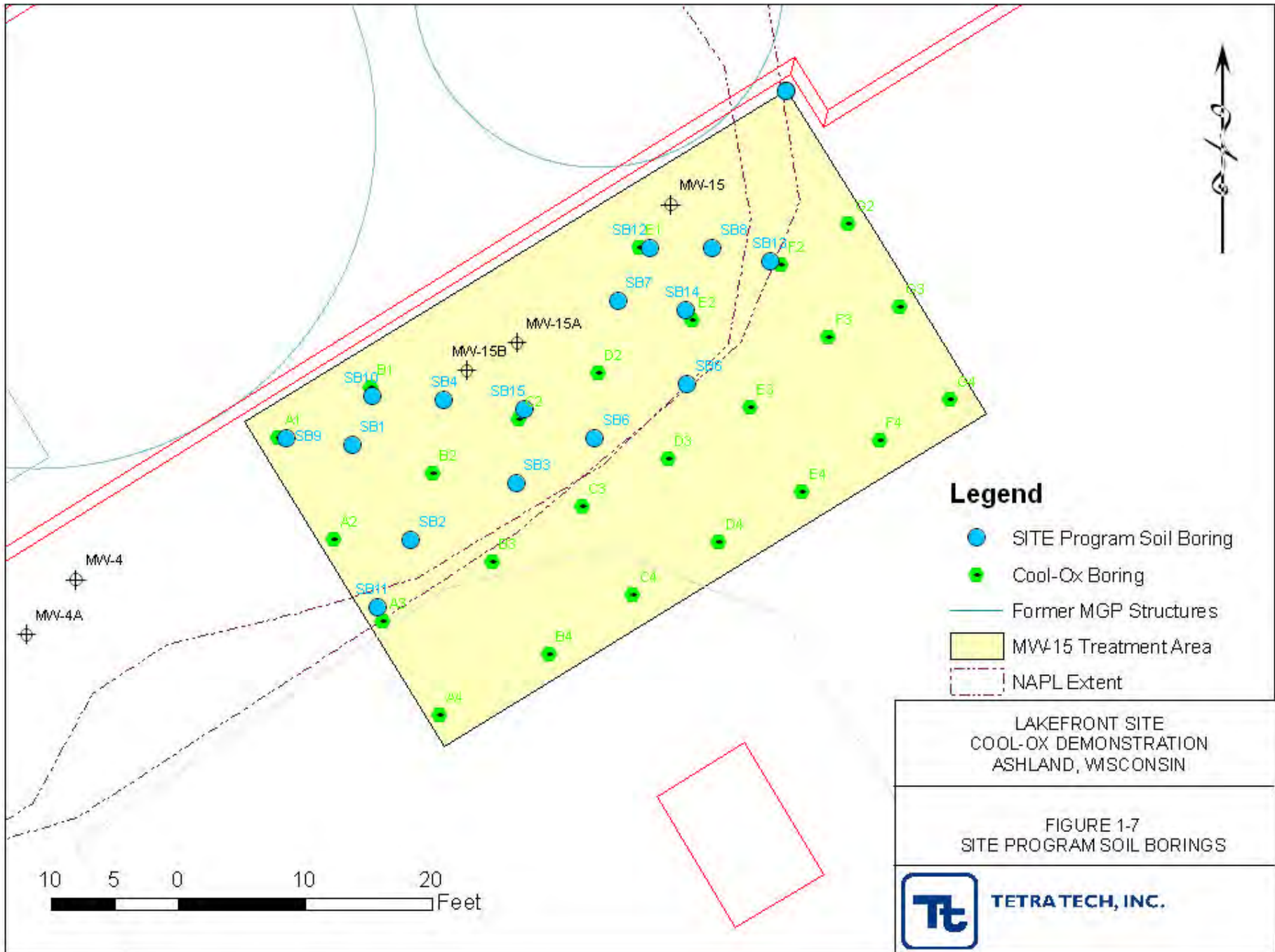
LAKEFRONT SITE
 COOL-OX DEMONSTRATION
 ASHLAND, WISCONSIN

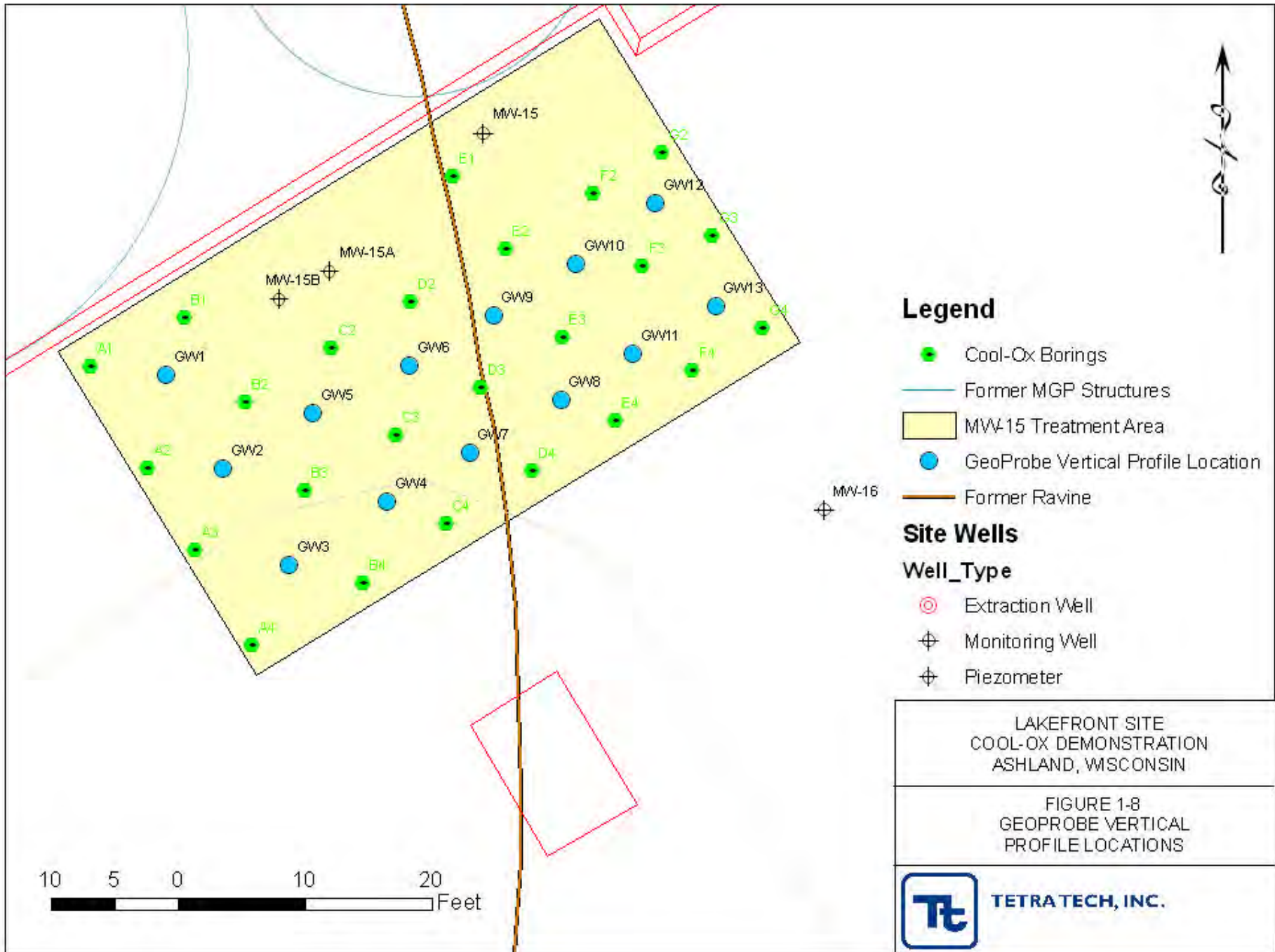
FIGURE 1-4
 EXTENT OF NAPL











Legend

- Cool-Ox Borings
- Former MGP Structures
- MVV-15 Treatment Area
- GeoProbe Vertical Profile Location
- Former Ravine

Site Wells

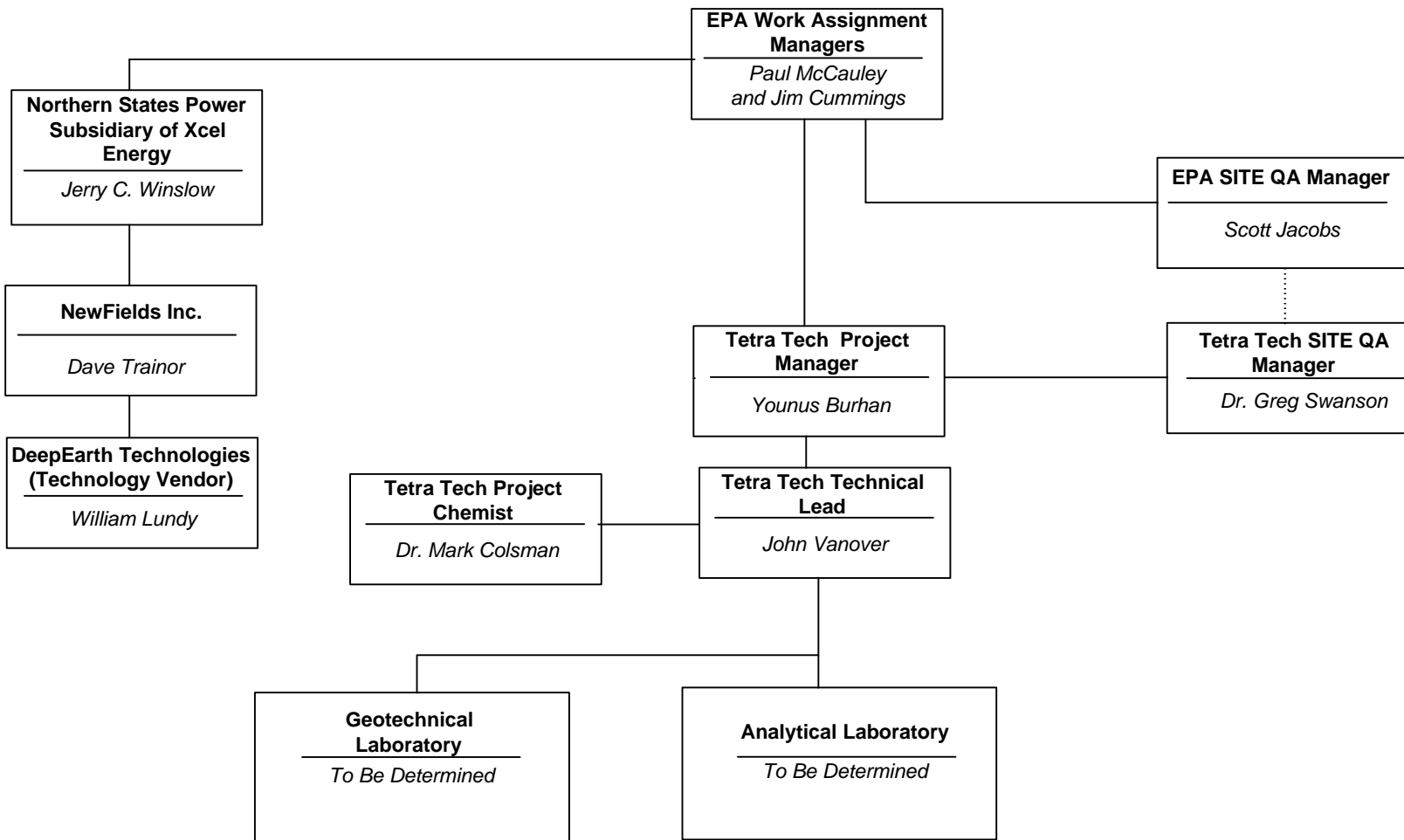
Well_Type

- ⊙ Extraction Well
- ⊕ Monitoring Well
- ⊕ Piezometer

LAKEFRONT SITE
 COOL-OX DEMONSTRATION
 ASHLAND, WISCONSIN

FIGURE 1-8
 GEOPROBE VERTICAL
 PROFILE LOCATIONS





LAKEFRONT SITE
COOL-OX DEMONSTRATION
ASHLAND, WISCONSIN

FIGURE 2-1
ORGANIZATIONAL CHART



Notes:
WAM - Work Assignment Manager
QA/QC - Quality Control & Quality Assurance

TABLES
(17 Pages)

TABLE 1-1 ⁽¹⁾
SUMMARY OF CONTAMINANTS IN RAVINE SOIL
COOL-OX™ DEMONSTRATION
Ashland, Wisconsin

Parameters	Number of Analyses	Number of Detections ⁽²⁾	Frequency of Detection	Maximum Detection	Minimum Detection	Average Detection ⁽³⁾
Ethylbenzene	115	103	90%	170000	2.5	5487
Benzo(a)pyrene	115	114	99%	340000	10	13665
Benzene	115	114	99%	230000	2.5	9472
Napththalene	115	115	100%	2900000	6.5	104395
Phenanthrene	115	114	99%	3700000	10	98670
Pyrene	115	114	99%	2000000	10	55822
Toluene	115	114	99%	320000	2.5	9536
Xylenes	115	24	21%	320000	2.5	27565

Notes:

- 1 - Analytical results were provided by Newfields in an electronic database. All results are in µg/Kg.
- 2 - Non detects were not specifically identified in the database. Blank spaces were assumed to indicate non detect.
- 3 - Non detects were not included in the calculation.

TABLE 1-2⁽¹⁾
SUMMARY OF CONTAMINANTS IN RAVINE GROUNDWATER
COOL-OX™ DEMONSTRATION
Ashland, Wisconsin

Parameters	Number of Analyses	Number of Detections⁽²⁾	Frequency of Detection	Maximum Detection	Minimum Detection	Average Detection⁽³⁾
Benzo(a)pyrene	107	36	34%	71000	0.03	1300
Benzene	107	76	71%	86000	0.27	7698
Ethylbenzene	107	57	53%	3400	0.19	743
Naphthalene	107	83	78%	1700000	0.41	33912
Phenanthrene	107	54	50%	390000	0.81	7180
Pyrene	107	46	43%	180000	0.11	3498
Toluene	107	66	62%	36000	0.11	3279
Xylenes	107	35	33%	10000	0.61	1236

Notes:

- 1 - Analytical results were provided by Newfields in an electronic database. All results are in µg/L.
- 2 - Non detects were not specifically identified in the database. Blank spaces and zeros were assumed to indicate non detect.
- 3 - Non detects were not included in the calculation.

TABLE 1-3
CONTAMINANTS OF CONCERN
COOL-OX™ DEMONSTRATION
Ashland, Wisconsin

Parameters	Fraction
Benzo(a)pyrene	Polynuclear Aromatic Hydrocarbon
Napthalene	Polynuclear Aromatic Hydrocarbon
Phenanthrene	Polynuclear Aromatic Hydrocarbon
Pyrene	Polynuclear Aromatic Hydrocarbon
Benzene	Volatile Organic Compound
Ethylbenzene	Volatile Organic Compound
Toluene	Volatile Organic Compound

TABLE 1-4
COOL-OX DEMONSTRATION SCHEDULE
Ashland, Wisconsin

Task	Description	Date
Baseline Sampling	Collect groundwater samples at MW-15 and MW-15A	October 23- November 10, 2006
	Collect soil samples in MW-15 area	November 6 - 10, 2006
	Measure NAPL recovery and collect NAPL samples	October 23 - November 28, 2006
MW-15 Treatment	Observe Cool-Ox Deployment in MW-15 area Continue NAPL monitoring	November 13 - 28, 2006
MW-13 Treatment	Inject Cool-Ox in MW-13 area	November 29, 2006 - January 2, 2007
Intermediate Sampling Event (30 days after treatment of MW-15 area)	Collect groundwater samples at MW-15 and MW-15A Collect soil samples in MW-15 area Conduct vertical profiling in MW-15 area	January 2, 2007 - January 12, 2007
Intermediate NAPL Monitoring event (30 days after MW-13 treatment is complete)	Measure NAPL recovery and collect NAPL samples	February 2, 2007
Final Sampling Event (90 days after treatment of MW-15 area)	Collect groundwater samples at MW-15 and MW-15A Collect soil samples in MW-15 area	March 5-9, 2007
Final NAPL Monitoring Event (90 days after treatment of MW-13)	Measure NAPL recovery and collect NAPL samples	April 2, 2007

**TABLE 3-1
SAMPLE SUMMARY
COOL-OX DEMONSTRATION
Ashland, Wisconsin**

Sample Location	Matrix	Parameters	Baseline ⁽¹⁾ Sample Event	Intermediate ⁽²⁾ Sample Event	Final ⁽³⁾ Sample Event	Total Samples	Analytical Method	Objective
SB1-SB15	Soil	SVOCs	45	45	45	135	SW-846 8270C	P1
		VOCs	45	45	45	135	SW-846 8260B	P1
		Metals	45	-	-	45	SW-846 6010B	S3
		Heterotrophic Plate Counts	22	22	22	66	SM9215B	S2
		PAH Degraders	22	22	22	66	SM9215B	
GW1-GW13	Groundwater	Bromide	0	26	0	26	EPA 300.0	S1
		Hydrogen Peroxide	0	26	0	26	Hach Field Kit	
MW-15	Groundwater	Heterotrophic Plate Counts	3	1	1	5	SM9215B	S2
		PAH Degraders	3	1	1	5	SM9215B	
		Nitrate	3	1	1	5	EPA 300.0	
		Sulfate	3	1	1	5	EPA 300.0	
		Total Organic Carbon	3	1	1	5	EPA415.1/SM5310	
		Total Inorganic Carbon	3	1	1	5	SM 2320	
		Biochemical Oxygen Demand	3	1	1	5	EPA 405.1	
		Chemical Oxygen Demand	3	1	1	5	EPA410.1	
		Dissolved Iron	3	1	1	5	Hach Field Kit	
		Dissolved Manganese	3	1	1	5	Hach Field Kit	
		Bromide	1	0	0	1	EPA 300.0	S1
		Metals	1	0	0	1	SW-846 6010B	S3
		SVOCs	3	1	1	5	SW-846 8270C	S4
		VOCs	3	1	1	5	SW-846 8260B	
				Temperature & pH	During each groundwater sampling event and for 7 days after Cool-Ox™ treatment of the MW-15 area.			
MW-15A	Groundwater	Heterotrophic Plate Counts	3	1	1	5	SM9215B	S2
		PAH Degraders	3	1	1	5	SM9215B	
		Nitrate	3	1	1	5	EPA 300.0	
		Sulfate	3	1	1	5	EPA 300.0	
		Total Organic Carbon	3	1	1	5	EPA415.1/SM5310	

**TABLE 3-1
SAMPLE SUMMARY
COOL-OX DEMONSTRATION
Ashland, Wisconsin**

Sample Location	Matrix	Parameters	Baseline ⁽¹⁾ Sample Event	Intermediate ⁽²⁾ Sample Event	Final ⁽³⁾ Sample Event	Total Samples	Analytical Method	Objective
MW-15A (Continued)	Groundwater	Total Inorganic Carbon	3	1	1	5	SM 2320	S2
		Biochemical Oxygen Demand	3	1	1	5	EPA 405.1	
		Chemical Oxygen Demand	3	1	1	5	EPA410.1	
		Dissolved Iron	3	1	1	5	Hach Field Kit	
		Dissolved Manganese	3	1	1	5	Hach Field Kit	
		Bromide	1	0	0	1	EPA 300.0	S1
		Metals	1	0	0	1	SW-846 6010B	S3
		SVOCs	3	1	1	5	SW-846 8270C	S4
		VOCs	3	1	1	5	SW-846 8260B	
Temperature & pH	During each groundwater sampling event and for 7 days after Cool-Ox™ treatment of the MW-15 area.				Field Meters	S6		
MW-13A	NAPL	Measure	3	1	1	5	Field Meter	S7
		Density	3	1	1	5	ASTM D1298-99	S8
		Viscosity	3	1	1	5	ASTM D7152-05	
		Surface Tension	3	1	1	5	ASTM D3825	
		SVOCs	0	0	0	0	SW-846 8270C	
		VOCs	0	0	0	0	SW-846 8260B	
		TPH-Extractables	0	0	0	0	SW-846 8015 Modified	
Temperature & pH	During each groundwater sampling event and for 7 days after Cool-Ox™ treatment of the MW-13 area.				Field Meters	S6		
EW-1	NAPL	Measure	3	1	1	5	Field Meter	S7
		Density	3	1	1	5	ASTM D1298-99	S8
		Viscosity	3	1	1	5	ASTM D7152-05	
		Surface Tension	3	1	1	5	ASTM D3825	
		SVOCs	0	0	0	0	SW-846 8270C	
		VOCs	0	0	0	0	SW-846 8260B	
TPH-Extractables	0	0	0	0	SW-846 8015 Modified			

**TABLE 3-1
SAMPLE SUMMARY
COOL-OX DEMONSTRATION
Ashland, Wisconsin**

Sample Location	Matrix	Parameters	Baseline ⁽¹⁾ Sample Event	Intermediate ⁽²⁾ Sample Event	Final ⁽³⁾ Sample Event	Total Samples	Analytical Method	Objective
EW-2	NAPL	Measure	3	1	1	5	Field Meter	S7 S8
		Density	3	1	1	5	ASTM D1298-99	
		Viscosity	3	1	1	5	ASTM D7152-05	
		Surface Tension	3	1	1	5	ASTM D3825	
		SVOCs	0	0	0	0	SW-846 8270C	
		VOCs	0	0	0	0	SW-846 8260B	
		TPH-Extractables	0	0	0	0	SW-846 8015 Modified	
EW-3	NAPL	Measure	3	1	1	5	Field Meter	S7 S8
		Density	3	1	1	5	ASTM D1298-99	
		Viscosity	3	1	1	5	ASTM D7152-05	
		Surface Tension	3	1	1	5	ASTM D3825	
		SVOCs	0	0	0	0	SW-846 8270C	
		VOCs	0	0	0	0	SW-846 8260B	
		TPH-Extractables	0	0	0	0	SW-846 8015 Modified	
Treatment System	NAPL	Measure	3	1	1	5	Field Meter	S7 S8
		Density	3	1	1	5	ASTM D1298-99	
		Viscosity	3	1	1	5	ASTM D7152-05	
		Surface Tension	3	1	1	5	ASTM D3825	
		SVOCs	1	1	1	3	SW-846 8270C	
		VOCs	1	1	1	3	SW-846 8260B	
		TPH-Extractables	1	1	1	3	SW-846 8015 Modified	
MW-15 Area	Observations of technology Cool-Ox deployment and data collected for other objectives will be used to evaluate this objective.							S5

Notes:

- 1 - Baseline sampling will be conducted prior to Cool-Ox™ treatment.
2 - Intermediate sampling will occur approximately 30 days after Cool-Ox™ treatment.
3 - The final sampling event will occur approximately 90 days after Cool-Ox™ treatment.
TPH - Total petroleum hydrocarbons
VOCs - Volatile organic compounds
SVOCs - Semivolatile organic compounds

ASTM - American society of testing and materials
EPA - U.S. Environmental Protection Agency
SM - Standard methods for examination of water and wastewater
SW-846 - Test methods for evaluating solid waste, physical/chemical methods

TABLE 3-2
SOIL DATA FROM THE MW-15 AREA USED FOR STATISTICAL CALCULATIONS
COOL-OX DEMONSTRATION
Ashland, Wisconsin

Location ID	Upper Depth	Lower Depth	Ethyl Benzene	Benzo(a)-Pyrene	Benzene	Naphthalene	Phenanthrene	Pyrene	Toluene
GP122	6	8	12000	340000	50000	270000	3700000	2000000	3100
GP125	9	11	40000	16000	11000	700000	110000	50000	29000
GP125	12	14	2500	425	9100	41000	4100	2100	13000
GP127	17	18	1100	440	7400	12000	3600	1500	8900
GP128	4	6	6600	6800	650	100000	66000	25000	1100
GP128	10	12	7.5	17	7.5	15.5	18.5	18	5.5
GP130	4	6	35	260	130	210	410	630	510
GP131	6	8	8900	1700	2800	28000	10000	4800	550
GP131	10	12	110	41	42	570	380	150	12
Mean			7920	40600	9010	128000	433000	232000	6240
Std. Dev.			12800	112000	16000	231000	1230000	663000	9680
Geo. Mean			1100	1090	1120	9220	6080	3420	842

Notes:

Depths are in feet below ground surface.

Concentrations are in milligrams per kilogram (parts per billion).

TABLE 4-1
ANALYTICAL METHODS, MINIMUM SAMPLE VOLUMES, PRESERVATION REQUIREMENTS AND HOLDING TIMES
COOL-OX™ DEMONSTRATION
Ashland, Wisconsin

Parameter	Analytical Method	Extraction Method	Cleanup Method	Minimum Quantity	Holding Times		Container	Preservation
					Extraction	Analysis ⁽³⁾		
Soil								
SVOCs	SW-846 8270C	SW-846 3510C	SW-846 3630C ⁽⁴⁾	200 g	14 days	40 days	250-mL amber glass	Cool to 4°C
Volatile Organic Compounds	SW-846 8260B	SW-846 5035	NA	3 X 5 g	NA	14 days	3 Encore Samplers	MeOH or NaHSO ₄ , cool to 4°C
TAL Metals	SW-846 6010B	SW-846 3050B	NA	10 g	6 mo.	NA	250-mL glass	Cool to 4°C
Plate Counts ⁽²⁾	SM9215B/MPN	NA	NA	100 g	NA	24 hours	250-mL glass	Cool to 4°C
Groundwater								
SVOCs	SW-846 8270C	SW-846 3510C	SW-846 3611B ⁽⁴⁾	2 L	7 days	40 days	2 x 1-L amber glass	Cool to 4°C
Volatile Organic Compounds	SW-846 8260B	SW-846 5030	NA	2 X 40 mL	NA	14 days	2 x 40-mL vials	Cool to 4°C, pH<2 with HCl
TAL Metals	SW-846 6010B	SW-846 3010A	NA	200 mL	6 mo.	NA	500-mL polyethylene	Use HNO ₃ to adjust pH < 2
Anions (NO ₃ ⁻ /SO ₄ ²⁻)	EPA 300.0	NA	NA	50 mL	NA	48 hours	250 mL amber bottle	Cool to 4°C
Bromide	EPA 300.0	NA	NA	50 mL	NA	28 days	250 mL amber glass	Cool to 4°C
TOC	EPA 415.1/SM5310	NA	NA	2 x 200 mL	NA	28 days	2 x 250-mL amber glass	Cool to 4°C, pH<2 with HCl
Total Inorganic Carbon	EPA 415.1/SM5310	NA	NA	2 x 200 mL	NA	28 days	2 x 250-mL amber glass	Cool to 4°C
Biochemical Oxygen Demand	EPA 405.1	NA	NA	200 mL	NA	48 hours	250 mL amber glass	Cool to 4°C
Chemical Oxygen Demand	EPA 410.4	NA	NA	10 mL	NA	28 days	250 mL amber glass	Cool to 4°C
Plate Counts ⁽²⁾	SM9215B/MPN	NA	NA	500 mL	NA	24 hours	2 x 250-mL amber glass	Cool to 4°C

TABLE 4-1
ANALYTICAL METHODS, MINIMUM SAMPLE VOLUMES, PRESERVATION REQUIREMENTS AND HOLDING TIMES
COOL-OX™ DEMONSTRATION
Ashland, Wisconsin

Parameter	Analytical Method	Extraction Method	Cleanup Method	Minimum Quantity	Holding Times		Container	Preservation
					Extraction	Analysis ⁽³⁾		
NAPL								
Surface Tension	ASTM D971	NA	NA	100 mL	NA	NA	250 mL amber glass	None
Viscosity	ASTM D445	NA	NA	100 mL	NA	NA	250 mL amber glass	None
Density	ASTM D4052	NA	NA	100 mL	NA	NA	250 mL amber glass	None
SVOCs	SW-846 8270C	SW-846 3580A	SW-846 3611B ⁽¹⁾	100 mL	14 days	40 days	125-mL glass	Cool to 4°C
Volatile Organic Compounds	SW-846 8260B	SW-846 3580A or 5030/5035	NA	2 X 40 mL	NA	14 days	2 x 40-mL vials	Cool to 4°C
TPH-e ⁽¹⁾	SW-846 8015B	SW-846 3550B	SW-846 3660B	100 mL	14 days	40 days	125-mL glass	Cool to 4°C

Notes:

- 1) TPH-e methods will quantitate and report extractable hydrocarbons as diesel range and motor oil range organics.
 - 2) To include total heterotrophic counts as well as PAH degraders.
 - 3) Analysis holding times are from date of extractic
 - 4) Cleanup method SW-846 3611B and/or 3660B to be added as necessary to remove interferences for specific samples.
- °C degrees Celsius
- ASTM American Society for Testing Materials (www.astm.org)
- EPA U.S. Environmental Protection Agency (EPA) method from "Methods for the Chemical Analysis of Water and Wastes" (EPA-600/4-79-20).
- g gram
- L Liter
- mL milliliter
- MPN Most probable number techniques for hydrocarbon degraders adapted from "Methods for Soil Analysis. Part 2."
- SVOC Semivolatile organic compound
- SW-846 EPA method from "Test Methods for Evaluating Solid Waste" (www.epa.gov/sw-846).
- TPH-e Total petroleum hydrocarbons extractables

TABLE 4-2
QC SAMPLE SUMMARY
COOL-OX DEMONSTRATION
Ashland, Wisconsin

Revision: 1
Date: October 2006

Sample Location	Matrix ⁽¹⁾	Parameters	Number of Samples	Field Duplicate	Equipment Blank	MS/MSD	Total Samples
SB1-SB15	Soil	VOCs	135	9	9	9	162
		SVOCs	135	9	9	9	162
		Metals	45	3	3	-	51
		Heterotrophic Plate Counts	66	3	3	-	72
		PAH Degraders	66	3	3	-	72
GW1-GW13	Groundwater	Bromide	26	2	2	-	30
MW-15	Groundwater	Heterotrophic Plate Counts	5	3	-	-	8
		PAH Degraders	5	3	-	-	8
		Nitrate	5	3	-	-	8
		Sulfate	5	3	-	-	8
		Total Organic Carbon	5	3	-	-	8
		Total Inorganic Carbon	5	3	-	-	8
		Biochemical Oxygen Demand	5	3	-	-	8
		Chemical Oxygen Demand	5	3	-	-	8
		Metals	1	1	1	-	3
		Bromide	1	1	-	-	2
		SVOCs	5	3	1	1	10
		VOCs	5	3	1	1	10
MW-15A	Groundwater	Heterotrophic Plate Counts	5	-	-	-	5
		PAH Degraders	5	-	-	-	5
		Nitrate	5	-	-	-	5
		Sulfate	5	-	-	-	5
		Total Organic Carbon	5	-	-	-	5
		Total Inorganic Carbon	5	-	-	-	5
		Biochemical Oxygen Demand	5	-	-	-	5
		Chemical Oxygen Demand	5	-	-	-	5
		Metals	1	-	-	-	1
		Bromide	1	-	-	-	1
		SVOCs	5	-	-	-	5
				VOCs	5	-	-
MW-13A	DNAPL	Density	5	-	-	-	5
		Viscosity	5	-	-	-	5
		Surface Tension	5	-	-	-	5
EW-1	DNAPL	Density	5	-	-	-	5
		Viscosity	5	-	-	-	5
		Surface Tension	5	-	-	-	5
EW-2	DNAPL	Density	5	-	-	-	5
		Viscosity	5	-	-	-	5
		Surface Tension	5	-	-	-	5
EW-3	DNAPL	Density	5	-	-	-	5
		Viscosity	5	-	-	-	5
		Surface Tension	5	-	-	-	5

TABLE 4-2
QC SAMPLE SUMMARY
COOL-OX DEMONSTRATION
Ashland, Wisconsin

Revision: 1
 Date: October 2006

Sample Location	Matrix ⁽¹⁾	Parameters	Number of Samples	Field Duplicate	Equipment Blank	MS/MSD	Total Samples
Treatment System	DNAPL	Density	5	-	-	-	5
		Viscosity	5	-	-	-	5
		Surface Tension	5	-	-	-	5
		SVOCs	3	-	-	-	3
		VOCs	3	-	-	-	3
		TPH-Extractables	3	-	-	-	3

Notes:

(1) Quality control samples collected at a frequency of 5 percent for soil and 10 percent for groundwater

VOCs - Volatile organic compounds

SVOCs - Semivolatile organic compounds

TPH - Total petroleum hydrocarbons

DNAPL - Dense nonaqueous phase liquid

TABLE 6-1
QA OBJECTIVES FOR PRECISION, ACCURACY COMPLETENESS AND PROJECT REQUIRED REPORTING LIMITS
COOL-OX™ DEMONSTRATION
Ashland, Wisconsin

Parameter	Matrix	Analytical Method	Units	PRRL	Precision (%RPD)	Accuracy (%R)	Completeness ¹
VOCs	Soil	8260B	µg/kg	5	35	70-130	90%
SVOCs	Soil	8270C	µg/kg	330	35	70-130	90%
Metals	Soil	6010B	µg/kg	100 - 25,000	35	70-130	90%
SVOCs	Water	8270C	µg/L	10 - 50	30	50-150	75%
VOCs	Water	8260B	µg/L	5	30	75-125	75%
Metals	Water	6010B	µg/L	3 - 100	30	75-125	75%
Anions (NO ³⁻ /SO ⁴⁻)	Water	300.1	mg/L	0.5	30	75-125	75%
Bromide	Water	300.1	mg/L	0.5	30	75-125	90%
Total organic carbon	Water	415.1	mg/L	0.5	30	70-130	75%
Total inorganic carbon	Water	SM2320	mg/L	0.5	30	70-130	75%
Biochemical oxygen demand	Water	405.1	mg/L	2	30	70-130	75%
Chemical oxygen demand	Water	410.1	mg/L	2	30	70-130	75%
Plate count methods	Water and Soil	SM9215B	cfu/sample	NA	10	NA	75%
Extractable TPH	Water and NAPL	8015 Modified	µg/kg	50 - 200	35	60-140	75%
Surface tension	DNAPL	D3825	N/m	NA	20	1	75%
Viscosity	DNAPL	D7152-05	N s/m ²	NA	20	1	75%
Density	DNAPL	D1298-99	g/cm ³	NA	20	1	75%

1 - For soil and water samples, if fewer than 10 samples are collected, the completeness goal will be modified to reflect n-1 successfully collect-and analyzed samples.

VOC - Volatile organic compounds

SVOC - Semivolatile organic compounds

PAH - Polynuclear organic compounds

TPH - Total petroleum hydrocarbons

µg/L - Micrograms per liter

mg/L - Milligrams per liter

cfu - Colony forming unit

DNAPL - Dense onaqueous phase liquid

µg/kg - Micrograms per kilogram

m²/sec - Meters squared per second

g/cm³ - Grams per cubic centimeter

N/m - Newtons per meter

TABLE 6-2
SUMMARY OF QC LABORATORY PROCEDURES FOR COCs
COOL-OX™ DEMONSTRATION
Ashland, Wisconsin

Parameter	Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SVOCs	SW-846 8270C	GC/MS Tuning	Prior to initial calibration and calibration verifications	See Method 8270C	Tune instrument and recheck tuning
		Initial Calibration	At beginning of project, if a continuing calibration fails, if the system has undergone maintenance, if a new column is installed, or as needed	1. All COCs must have a mean RF of > 0.05% 2. RSD < 20% over minimum of 5 calibration standards for all COCs.	1. Terminate analysis 2. Perform system maintenance and recalibrate
		Calibration Verification	At beginning of each 12-hour period during which samples are analyzed	1. All COCs must have a mean RF of > 0.05% 2. % difference between RF and mean RF from initial calibration must be < 25% for all COCs.	1. Terminate analysis 2. Perform system maintenance and recalibrate
		Matrix Spike/ Matrix Spike Duplicate	With each batch of 20 or fewer samples	1. 60-140% Recovery 2. RPD < 35%	1. Notify Tetra Tech 2. Flag positive result in unspiked sample if recovery > 140% 3. Flag positive result or reporting limit in unspiked sample if recovery < 30%
		LCS	With each preparation batch of 20 or fewer samples	60-140% Recovery	1. Investigate and correct cause of problem 2. Re-analyze all samples in batch

TABLE 6-2
SUMMARY OF QC LABORATORY PROCEDURES FOR COCs
COOL-OX™ DEMONSTRATION
Ashland, Wisconsin

Parameter	Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SVOCs (continued)	SW-846 8270C (continued)	Surrogates	Added to each sample, blank QC sample, and standard	20-130% Recovery	Re-analyze any sample in which one or more surrogate compounds are outside specified limits
		Internal Standard Areas	All samples, blanks, and QC samples	Within +100% or -50% of area in calibration verification standard (or mid-point standard of initial calibration, if no calibration verification analyzed)	1. Investigate and correct cause of problem 2. Re-analyze all samples that do not meet criterion
		Method Blank	With each calibration verification or initial calibration	No COCs detected at or above the PRRL	Re-analyze all samples in batch with COCs detected at or above the PRRL
		Petroleum Ref. Material	With each calibration verification or initial calibration	50-150% of reference value	1. Investigate and correct problem 2. Reanalyze all samples in batch
VOCs	SW-846 8260B	GC/MS Tuning	Prior to initial calibration and calibration verifications	See Method 8260B	Tune instrument recheck tuning
		Initial Calibration	At beginning of project, if a continuing calibration fails, if the system has undergone maintenance, if a new column is installed, or as needed	1. All COCs must have a mean RF of > 0.05% 2. RSD < 30% over minimum of 5 calibrations standards (or coefficient of correlation (r) > 0.99) for all COCs.	Perform system maintenance and recalibrate

TABLE 6-2
SUMMARY OF QC LABORATORY PROCEDURES FOR COCs
COOL-OX™ DEMONSTRATION
Ashland, Wisconsin

Parameter	Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
VOCs (continued)	SW-846 8260B	Calibration Verification	At beginning of each 12-hour period during which samples are analyzed	1. All COCs must have a mean RF of > 0.05% 2. % difference between RF and mean RF must be < 30% (or % difference between known and measured value must be < 20% if linear regression used for calibration)	1. Terminate analysis 2. Perform system maintenance and recalibrate
		Matrix Spike/ Matrix Spike Duplicate	With each batch of 20 or fewer samples	1. 70 - 130% Recovery 2. RPD < 35%	1. Notify Tetra Tech 2. Flag positive result in unspiked sample if recovery > 130% 3. Flag positive result or reporting limit in unspiked sample if recovery < 70%
		LCS	With each preparation batch of 20 or fewer samples	75 - 125% Recovery	1. Investigate and correct cause of problem 2. Re-analyze all samples in batch
		Surrogates	Added to each sample, blank, QC sample and standard	Dibromofluoromethane. . . . 80-120% Recovery 1,2-Dichloroethane-d4 70-120% Recovery Toluene-d8.81-117% Recovery p-Bromofluorobenzene. . . . 74-121% Recovery	Re-analyze any sample in which one or more surrogate compounds are outside of specified limits.
		Internal Standard Areas	All samples, blanks, and QC Samples	Within +100% or -50% of area in calibration verification standard (or mid-point standard of initial calibration, if no calibration verification analyzed)	1. Investigate and correct cause of problem 2. Re-analyze all samples that do not meet criterion

TABLE 6-2
SUMMARY OF QC LABORATORY PROCEDURES FOR COCs
COOL-OX™ DEMONSTRATION
Ashland, Wisconsin

Parameter	Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
VOCs (continued)	SW-846 8260B	Method Blank	With each calibration verification or initial calibration	No COCs detected at or above the PRRL	Re-analyze all samples in batch with COCs detected at or above the PRRL

Notes and Abbreviations:

- COC Chemical of concern
- GC/MS Gas chromatography/mass spectrometry
- LCS Laboratory control sample
- PAH Polynuclear aromatic hydrocarbons
- PRRL Project-required reporting limit
- QC Quality control
- RF Response factor
- RPD Relative percent difference
- RSD Relative standard deviation
- SIM Selected ion monitoring
- SW-846 EPA's Test Methods for Evaluation Solid Waste
- VOC Volatile organic compounds

APPENDIX A

**COOL-OX™ DEMONSTRATION
HEALTH AND SAFETY PLAN
ASHLAND, WISCONSIN**



Site Name: Lakefront Site	Site Contact: John Vanover - TTEMI	Telephone: (513) 564-8352												
Location: 301 Lake Shore Drive East, Ashland, WI	Client Contact:	Telephone:												
EPA I.D. No. WISFN057952	Prepared By: B. Schroeder	Date: 9-7-2006												
Project No. G9015.3.065.02.01	Date of Activities: Intermittent from 10-01-06 through March 2006													
<p>Objectives: Tetra Tech will conduct a technology evaluation study at the Ashland/NSP Lakefront site. The investigation will include 5 to 12 sampling events over an approximately 5 month periods. Activities include collection of soil and groundwater samples.</p>	<p>Site Type: Check as many as applicable.</p> <table> <tr> <td><input type="checkbox"/> Active</td> <td><input type="checkbox"/> Landfill</td> <td><input type="checkbox"/> Residential</td> </tr> <tr> <td><input type="checkbox"/> Inactive</td> <td><input type="checkbox"/> Railroad</td> <td><input checked="" type="checkbox"/> Industrial</td> </tr> <tr> <td><input type="checkbox"/> Secured</td> <td><input type="checkbox"/> Uncontrolled</td> <td><input type="checkbox"/> Urban</td> </tr> <tr> <td><input type="checkbox"/> Unsecured</td> <td><input checked="" type="checkbox"/> Controlled</td> <td><input type="checkbox"/> Other (<i>specify</i>)</td> </tr> </table>		<input type="checkbox"/> Active	<input type="checkbox"/> Landfill	<input type="checkbox"/> Residential	<input type="checkbox"/> Inactive	<input type="checkbox"/> Railroad	<input checked="" type="checkbox"/> Industrial	<input type="checkbox"/> Secured	<input type="checkbox"/> Uncontrolled	<input type="checkbox"/> Urban	<input type="checkbox"/> Unsecured	<input checked="" type="checkbox"/> Controlled	<input type="checkbox"/> Other (<i>specify</i>)
<input type="checkbox"/> Active	<input type="checkbox"/> Landfill	<input type="checkbox"/> Residential												
<input type="checkbox"/> Inactive	<input type="checkbox"/> Railroad	<input checked="" type="checkbox"/> Industrial												
<input type="checkbox"/> Secured	<input type="checkbox"/> Uncontrolled	<input type="checkbox"/> Urban												
<input type="checkbox"/> Unsecured	<input checked="" type="checkbox"/> Controlled	<input type="checkbox"/> Other (<i>specify</i>)												
<p>Initial Site information</p> <p>The Ashland/NSP Lakefront Superfund Site (the "Site") consists of approximately 20 acres of affected land located on the shore of Chequamegon Bay of Lake Superior, in Ashland, Wisconsin. The Site contains property owned by Northern States Power Company, a Wisconsin corporation (d.b.a. Xcel Energy, a subsidiary of Xcel Energy, Inc. ("NSPW"), a portion of Kreher Park, a City owned property fronting on the bay, the former City Waste Water Treatment Plant (WWTP), also located at Kreher Park, and an inlet area containing contaminated sediment directly offshore from the former WWTP.</p> <p>From approximately 1885 to 1947, gas was generated for heating and lighting at a former manufactured gas plant (MGP) located at the NSP property. Manufactured gas plant wastes containing hazardous substances were released during the gas manufacturing process at the former MGP. The former MGP property was transected on the north by a ravine that ended at the historic shoreline of Chequamegon Bay. Historical maps show that the ravine was open at the startup of gas production at the former MGP in the late 1880s and was filled by the early 1900s.</p> <p>Site assessments and investigations conducted at the Site by NSPW, WDNR, and USEPA have identified high levels of coal tar and other waste materials in groundwater, soil and sediment throughout the Site. Manufactured gas plant waste contains hazardous substances, including volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), and polycyclic aromatic compounds (PAHs). Hazardous substances, including VOCs, SVOCs, and PAHs are present in an aquifer underneath the former MGP, in soil and a former seep area in Kreher Park, and in sediments in Chequamegon Bay.</p>														
Wind Speed and Direction (Approach from upwind):	Temperature (°F):	Precipitation: Forecast: Precipitation:												

Note: A site sketch is provided on Page 5 of 12.



LEVEL-TWO HEALTH AND SAFETY PLAN

Initial Isolation and Protective Action Distances: Use the A2000 Emergency Response Guidebook® (ERG)

Initial Isolation Distance: This zone should extend in all directions; 660 feet for unknown hazards and 0.5 mile for tanker truck or rail car incidents.

NOTE: Keep a maximum distance away for unknown sites until the identity of the material(s) are determined.

Subsequent Isolation and Protection Action Zones Based on Air Monitoring Results:

NOTE: Distance at sites with unknown hazards should be increased, if necessary, based on air monitoring results.

Waste Type: [X] Liquid [X] Solid [] Sludge [] Gas [] Unknown

Waste Characteristics: Field screening and/or Hazcat™ test may be used to identify if product-specific information is not available. Check as many as applicable.

- Corrosive, Toxic, Inert, Ignitable, Flammable, Volatile, Reactive, Radioactive, Unknown, Peroxide forming, Other (specify)

Hazard(s) of Concern: Check as many as applicable.

- Heat stress, Cold stress, Explosion or fire hazard, Oxygen deficiency, Radiological hazard, Underground storage tanks, Surface tanks, Buried utilities, Overhead utilities, Confined space(s), Noise, Biological hazard, Inorganic chemicals, Organic chemicals, Heavy equipment, Other (specify)

Explosion or Fire Potential: [] High [] Medium [X] Low [] Unknown



Chemical Products Tetra Tech EM Inc. Will Use or Store On Site: (Attach a Material Safety Data Sheet [MSDS] for each item.)

- | | | | |
|---|--|--|---|
| <input type="checkbox"/> Alconox7 or Liquinox7 | <input type="checkbox"/> Calibration gas (Methane) | <input type="checkbox"/> Hexane | <input type="checkbox"/> Isopropyl alcohol |
| <input checked="" type="checkbox"/> Hydrochloric acid (HCl) | <input type="checkbox"/> Calibration gas (Isobutylene) | <input type="checkbox"/> Household bleach (NaOCl) | <input type="checkbox"/> HazcatJ Kit |
| <input type="checkbox"/> Nitric acid (HNO ₃) | <input type="checkbox"/> Calibration gas (Pentane) | <input type="checkbox"/> Sulfuric acid (H ₂ SO ₄) | <input type="checkbox"/> Mark I Kits (<i>number?</i>) _____ |
| <input type="checkbox"/> Sodium hydroxide (NaOH) | <input type="checkbox"/> Hydrogen gas | <input type="checkbox"/> Acetic acid | <input type="checkbox"/> Other (<i>specify</i>) _____ |

Applicable Safe Work Practices (SWP) attach to ERHASP:

Check as many as applicable

- SWP 6-1 - General Safe Work Practices
- SWP 6-2 - Control of Hazardous Energy Sources (Lockout/Tagout)
- SWP 6-3 - Safe Drilling Practices
- SWP 6-4 - Excavation Practices
- SWP 6-5 - Working Over or Near Water
- SWP-6-6 - Hot Work Practices
- SWP 6-7 - Special Site Hazards
- SWP 6-8 - Safe Electrical Work Practices
- SWP 6-9 - Fall Protection Practices
- SWP 6-10 - Portable Ladder Safety
- SWP 6-11 - Drum and Container Handling Practices
- SWP 6-12 - Shipping Dangerous Goods
- SWP 6-13 - Flammable Hazards and Ignition Sources
- SWP 6-14 - Spill and Discharge Control Practices
- SWP 6-15 - Heat Stress
- SWP 6-16 - Cold Stress
- SWP 6-17 - Biohazards
- SWP 6-18 - Underground Storage Tank Removal Practices
- SWP 6-19 - Working Safely with Hydrazine
- SWP 6-20 - Working Safely with Benzene
- SWP 6-21 - Radiation Safety Practices
- SWP 6-22 - Hydrographic Data Collection
- SWP 6-23 - Permit-Required Confined Space
- SWP 6-24 - Non-Permit-Required Confined Space
- SWP 6-25 - Oil and Petroleum Distillate Fuel Product Hazards
- SWP 6-26 - Use of Heavy Equipment
- SWP 6-27 - Respirator Cleaning Procedures
- SWP 6-28 - Safe Work Practices for Use of Air Purifying Respirators
- SWP 6-29 - Respirator Qualitative Fit Testing Procedures
- SWP 6-32 - Safe Work Practice for Sampling Anthrax Contamination in Buildings

Tetra Tech Employee Training and Medical Requirements:

Basic Training and Medical

- Initial 40 Hour Training
- 8-Hour Supervisor Training (one-time)
- Current 8-Hour Refresher Training
- Current Medical Clearance (including respirator use)
- Current First Aid Training (minimum 1 Tetra Tech employee on site)
- Current CPR Training (minimum 1 Tetra Tech employee on site)

Other Specific Training

- Confined Space Training
- Level A Training
- Radiation Training
- Atropine (Nerve Agent Antidote) Injector Training
- Other _____



LEVEL-TWO HEALTH AND SAFETY PLAN

Materials Present or Suspected at Site	Highest Observed Concentration (specify units and media)	Exposure Limit (specify ppm or mg/m ³)	IDLH Level (specify ppm or mg/m ³)	Primary Hazards of the Material (explosive, flammable, corrosive, toxic, volatile, radioactive, biohazard, oxidizer, etc.)	Symptoms and Effects of Acute Exposure	Photo-ionization Potential (eV)
Coal Tar Pitch Volatiles, PAH's	6,676 ppm Soil 107 ppm Water	PEL = 0.2 mg/m ³ TWA REL = 0.1 mg/m ³ TLV = 0.2 mg/m ³ TWA	80 mg/m ³	Decomposes on heating above 400°C producing toxic fumes . Reacts with strong oxidants .	Irritating to the eyes , the skin and the respiratory tract .Exposure to sun may enhance the irritating effect of coal tar pitch on skin and eyes and lead to burns.	
PAH's	6,676 PPM Soil 694 PPM Water	PEL = 0.2 mg/m ³ REL = TLV =	80 mg/m ³	volatile		
Benzene	643 ppm Soil 790 ppm Water	PEL =1ppm TWA REL = 0.1ppm TWA TLV =	500 ppm	volatile	Irritation to eyes, skin, nose, respiratory system; dizziness; headache, nausea, staggered gait; anorexia, lassitude (weakness, exhaustion);	9.24 eV
Ethyl Benzene	2,973 ppm Soil 5.3 PPM Water	PEL =100ppm TWA REL = 100ppm TWA TLV =	800 ppm	volatile	Irritation eyes, skin, mucous membrane; headache; dermatitis; narcosis, coma	8.76 eV
Toluene	2,007 ppm Soil 36 ppm Water	PEL =200ppm TWA REL = 100ppm TWA TLV =	500 ppm	volatile	Irritation eyes, nose; lassitude (weakness, exhaustion), confusion, euphoria, dizziness, headache; dilated pupils, lacrimation (discharge of tears	8.82 eV
Xylene	4,981 ppm Soil 10 PPM Water	PEL = 100 ppm TWA REL = 100 ppm TLV =	900 ppm	volatile	Irritation eyes, skin, nose, throat; dizziness, excitement, drowsiness, incoordination, staggering gait	8.56 eV
Naphthalene	2,900 ppm Soil	PEL =10 ppm TWA REL = 10 ppm TWA TLV =	300 ppm	volatile	Irritation eyes; headache, confusion, excitement, malaise (vague feeling of discomfort); nausea, vomiting, abdominal pain	8.12 eV
		PEL = REL = TLV =				

Information Source(s): NIOSH Pocket Guide to Chemical Hazards

Note: Use the following short forms to complete the table above.

A = Air
CARC = Carcinogenic
eV = Electron volt
GW = Groundwater

IDLH = Immediately dangerous to life or health
mg/m³ = Milligram per cubic meter
NA = Not available
NE = None established

PEL = Permissible exposure limit
ppm = Part per million
REL = Recommended exposure limit
S = Soil

SW = Surface water
TLV = Threshold limit value
U = Unknown



LEVEL-TWO HEALTH AND SAFETY PLAN

Field Activities Covered Under This Plan:									
Task Description	Level of Protection ¹								Date of Activities
	Primary				Contingency				
1 Soil Boring/sampling	<input type="checkbox"/> A	<input type="checkbox"/> B	<input type="checkbox"/> C	<input checked="" type="checkbox"/> D	<input type="checkbox"/> A	<input type="checkbox"/> B	<input checked="" type="checkbox"/> C	<input type="checkbox"/> D	
2 Groundwater sampling	<input type="checkbox"/> A	<input type="checkbox"/> B	<input type="checkbox"/> C	<input checked="" type="checkbox"/> D	<input type="checkbox"/> A	<input type="checkbox"/> B	<input checked="" type="checkbox"/> C	<input type="checkbox"/> D	
3	<input type="checkbox"/> A	<input type="checkbox"/> B	<input type="checkbox"/> C	<input type="checkbox"/> D	<input type="checkbox"/> A	<input type="checkbox"/> B	<input type="checkbox"/> C	<input type="checkbox"/> D	
4	<input type="checkbox"/> A	<input type="checkbox"/> B	<input type="checkbox"/> C	<input type="checkbox"/> D	<input type="checkbox"/> A	<input type="checkbox"/> B	<input type="checkbox"/> C	<input type="checkbox"/> D	
5	<input type="checkbox"/> A	<input type="checkbox"/> B	<input type="checkbox"/> C	<input type="checkbox"/> D	<input type="checkbox"/> A	<input type="checkbox"/> B	<input type="checkbox"/> C	<input type="checkbox"/> D	
Site Personnel and Responsibilities (include subcontractors):									
Employee Name and Office Code	Task(s)	Responsibilities							
John Vanover	1,2	<ul style="list-style-type: none"> Project Manager or Field Team Leader: Directs project investigation activities, makes site safety coordinator (SSC) aware of pertinent project developments and plans, and maintains communications with client as necessary. Site Safety Coordinator (SSC): Ensures that appropriate personal protective equipment (PPE) is available, enforces proper utilization of PPE by on-site personnel, suspends investigative work if he or she believes that site personnel are or may be exposed to an immediate health hazard, implements the health and safety plan, and reports any observed deviations from anticipated conditions described in the health and safety plan to the health and safety representative. Field Personnel: Completes tasks as directed by the project manager, field team leader, and SSC, and follows all procedures and guidelines established in the Tetra Tech, Inc., Health and Safety Manual. 							
Dustin Reed	1,2								
Dustin Reed Brad Schroeder other	1,2								

Note: ¹ See next page for details regarding levels of protection



LEVEL-TWO HEALTH AND SAFETY PLAN

Protective Equipment: (Indicate type or material as necessary for each task.)				
Task	Primary Level of Protection (A,B,C,D)	PPE Component Description (Primary)	Contingency Level of Protection (A, B, C, D)	PPE Component Description (Contingency)
1	D	Respirator type: None Cartridge type (if applicable): CPC material: Tyvek Glove material(s): Nitrile Boot material: Steel toe Other: Hard hat	C	Respirator type: Cartridge type (if applicable): CPC material: Tyvek Glove material(s): Nitrile Boot material: Steel toe Other: Hard hat
2	D	Respirator type: Cartridge type (if applicable): CPC material: Tyvek Glove material(s): Boot material: Other:	C	Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:
3		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:
4		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:
5		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:		Respirator type: Cartridge type (if applicable): CPC material: Glove material(s): Boot material: Other:

Notes:

All levels of protection must include eye, head, and foot protection.

CPC = Chemical protective clothing

Thermoluminescent Dosimeter (TLD) Badges must be worn to all field activities. TLDs must be worn under CPC.



LEVEL-TWO HEALTH AND SAFETY PLAN

Monitoring Equipment: (Specify instruments needed for each task; attach additional sheets as necessary)				
Instrument	Task	Instrument Reading	Action Guideline	Comments
Combustible gas indicator model:	<input checked="" type="checkbox"/> 1	0 to 10% LEL	Monitor; evacuate if confined space	<input type="checkbox"/> Not needed
	<input checked="" type="checkbox"/> 2	10 to 25% LEL	Potential explosion hazard; notify SSC	
	<input type="checkbox"/> 3			
	<input type="checkbox"/> 4	>25% LEL	Explosion hazard; interrupt task; evacuate site; notify SSC	
	<input type="checkbox"/> 5			
Oxygen meter model:	<input checked="" type="checkbox"/> 1	>23.5% Oxygen	Potential fire hazard; evacuate site	<input type="checkbox"/> Not needed
	<input checked="" type="checkbox"/> 2	23.5 to 19.5% Oxygen	Oxygen level normal	
	<input type="checkbox"/> 3	<19.5% Oxygen	Oxygen deficiency; interrupt task; evacuate site; notify SSC	
	<input type="checkbox"/> 4			
	<input type="checkbox"/> 5			
Radiation survey meter model:	<input type="checkbox"/> 1	Normal background	Proceed	C Annual exposure not to exceed 1,250 mrem per quarter C Background reading must be taken in an area known to be free of radiation sources.
	<input type="checkbox"/> 2	Two to three times background	Notify SSC	
	<input type="checkbox"/> 3	>Three times background	Radiological hazard; interrupt task; evacuate site; notify Health Physicist	
	<input type="checkbox"/> 4			
	<input type="checkbox"/> 5			
Photoionization detector model: <input type="checkbox"/> 11.7 eV <input checked="" type="checkbox"/> 10.6 eV <input type="checkbox"/> 10.2 eV <input type="checkbox"/> 9.8 eV <input type="checkbox"/> _____ eV	<input checked="" type="checkbox"/> 1	Any response above background to 5 ppm above background	Level C ^a is acceptable Level B is recommended	C These action levels are for unknown gases or vapors. After the contaminants are identified, action levels should be based on the specific contaminants involved.
	<input checked="" type="checkbox"/> 2	> 5 to 500 ppm above background	Level B	
	<input type="checkbox"/> 3	> 500 ppm above background	Level A	
	<input type="checkbox"/> 4			
	<input type="checkbox"/> 5			
Flame ionization detector model:	<input type="checkbox"/> 1	Any response above background to 5 ppm above background	Level C ^a is acceptable Level B is recommended	C These action level are for unknown gases or vapors. After the contaminants are identified, action levels should be based on the specific contaminants involved.
	<input type="checkbox"/> 2	>5 to 500 ppm above background	Level B	
	<input type="checkbox"/> 3	>500 above background	Level A	
	<input type="checkbox"/> 4			
	<input type="checkbox"/> 5			
Detector tube models:	<input type="checkbox"/> 1	Specify: < 2 the PEL	Specify:	C The action level for upgrading the level of protection is one-half of the contaminants PEL. If the PEL is reached, evacuate the site and notify a safety specialist
	<input type="checkbox"/> 2	> 2 the PEL		
	<input type="checkbox"/> 3			
	<input type="checkbox"/> 4			
	<input type="checkbox"/> 5			
Other (specify):	<input type="checkbox"/> 1	Specify:	Specify:	<input checked="" type="checkbox"/> Not needed
	<input type="checkbox"/> 2			
	<input type="checkbox"/> 3			
	<input type="checkbox"/> 4			
	<input type="checkbox"/> 5			

Notes:

eV= electron volt

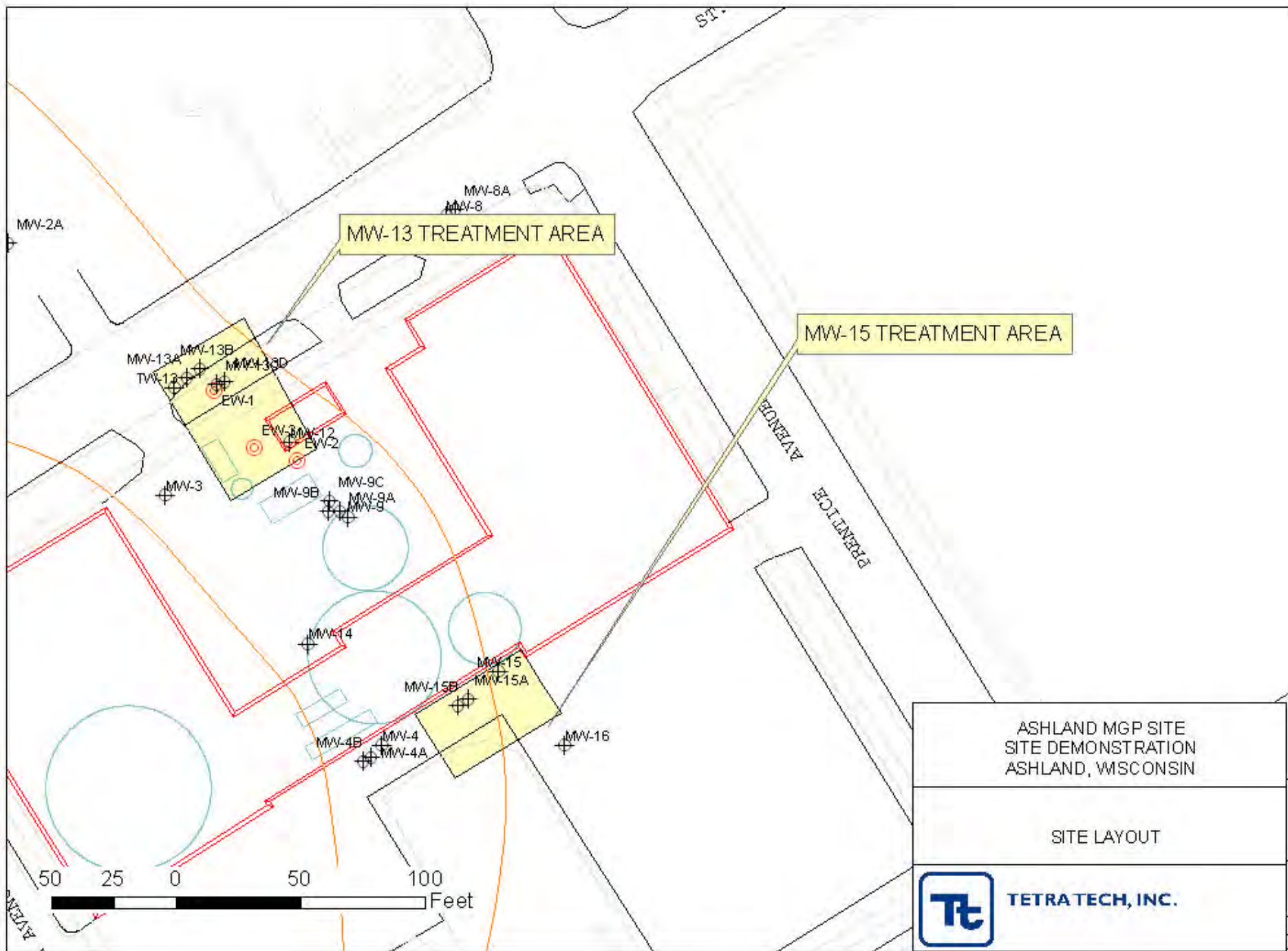
LEL=Lower explosive limit

mrem=Millirem

PEL=Permissible exposure limit

ppm=Part per million

^a Level C may be acceptable for certain tasks in some situations. If you are uncertain whether Level C is appropriate, consult the Regional Safety Officer. Additionally, when working with unknown respiratory hazards, Level C cartridge must provide protection for organic vapors, acid gases, ammonia, amines, formaldehyde, hydrogen fluoride, and particulate aerosols.



ST.

MW-13 TREATMENT AREA

MW-15 TREATMENT AREA

EDINBORO AVENUE
EDINBORO STREET



MW-13A MW-13B MW-13C
TW-13

EW-1

EW-3

MW-12

EW-2

MW-3

MW-9B

MW-9C

MW-9A

MW-9

MW-14

MW-15B

MW-15A

MW-4B

MW-4

MW-4A

MW-16

MW-2A

MW-8A

MW-8

EDINBORO AVENUE



APPROVAL AND SIGN-OFF FORM

Project No.: _____

I have read, understood, and agree with the information set forth in this Health and Safety Plan and will follow the direction of the Site Safety Coordinator as well as procedures and guidelines established in the Tetra Tech, Inc., Health and Safety Manual. I understand the training and medical requirements for conducting field work and have met these requirements

Name

Signature

Date

Name

Signature

Date

Name

Signature

Date

Name

Signature

Date

APPROVALS (Two Signatures Required):

Site Safety Coordinator

Date

Health and Safety Plan Reviewer/Approver

Date

Note:

Guidance in the ASTART Health and Safety Plan Approval Procedures, dated September 19, 2001, must be followed by personnel who prepare and approve any LEVEL-TWO HASP.